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Analytical Method Evaluation for Measuring Ethylene Oxide Emissions from Commercial Dilute-Acid Hydrolytic Control Units

Radian Corp., Research Triangle Park, NC

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ANALYTICAL METHOD EVALUATION FOR MEASURING ETHYLENE OXIDE EMISSIONS FROM COMMERCIAL DILUTE-ACID HYDROLYTIC CONTROL UNITS

By

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The Source Branch of the Environmental Protection Agency's (EPA) Environmental Monitoring Systems Laboratory (EMSL) at Research Triangle Park, North Carolina, has a program to develop stationary source test methods of known precision and accuracy for determining compliance with EPA standards. While participating in this program, Radian Corporation performed a field evaluation of a method for sampling and analyzing ethylene oxide (EO) in the vent stream from a dilute acid scrubber of a commercial sterilizer. The purposes of the test were to evaluate the ability of the analytical method to quantitate EO in scrubber emissions, to determine EO stability in sample containers, and to measure the EO concentration profile and EO-to-oxygen and EO-to-dichlorodifluoromethane (CFC-12) ratios during the initial sterilizer evacuation.

The test method acquired three-second grab samples that were analyzed by gas chromatography with flame ionization detection. Samples were acquired at one-minute, three-minute, four-minute or six-minute intervals. All samples were analyzed in the field and a portion of the samples were reanalyzed in the laboratory.

The analytical method was evaluated on three 1/8-inch (3 millimeter) OD stainless steel columns packed with a liquid phase coated on 60/80 mesh Carbopack B. The 5% Fluorcol column was 10 feet (3 meters) long; the 3% SP-1000 column was 8 feet long (2.4 m); and the 1% SP-1000 column was 6 feet (1.8 m) long. Evaluations based on column efficiency, resolution, linearity, retention time stability, and limits of (SEE ATTACHED SHEET)

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ABSTRACT

The Source Branch of the Environmental Protection Agency's (EPA) Environmental Monitoring Systems Laboratory (EMSL) at Research Triangle Park, North Carolina, has a program to develop stationary source test methods of known precision and accuracy for determining compliance with EPA standards. While participating in this program, Radian Corporation performed a field evaluation of a method for sampling and analyzing ethylene oxide (EO) in the vent stream from a dilute acid scrubber of a commercial sterilizer. The purposes of the test were to evaluate the ability of the analytical method to quantitate EO in scrubber emissions, to determine EO stability in sample containers, and to measure the EO concentration profile and EO-to-oxygen and EO-to-dichlorodifluoromethane (CFC-12) ratios during the initial sterilizer evacuation.

The test method acquired three-second grab samples that were analyzed by gas chromatography with flame ionization detection. Samples were acquired at one-minute, three-minute, four-minute or six-minute intervals. All samples were analyzed in the field and a portion of the samples were reanalyzed in the laboratory.

The analytical method was evaluated on three 1/8-inch (3 millimeter) OD stainless steel columns packed with a liquid phase coated on 60/80 mesh Carbopack B. The 5% Fluorcol column was 10 feet (3 meters) long; the 3% SP-1000 column was 8 feet long (2.4 m); and the 1% SP-1000 column was 6 feet (1.8 m) long. Evaluations based on column efficiency, resolution, linearity, retention time stability, and limits of detection and quantitation showed that a 5% Fluorocol column at 65°C was best for measuring percent level EO and CFC-12 concentrations and a 1% SP-1000 column at 45°C was best for measuring part-per-million EO levels. Under these conditions, baseline resolution of EO from CFC-12 was obtained. The 1% SP-1000 column showed good

retention time stability and can be used for both scrubber inlet and outlet monitoring.

Sample stability was evaluated in one-liter Tedlar bags, five-milliliter gas-tight syringes, and Vacu-Samplers. Results indicated that grab samples in gas-tight syringes should be analyzed within several hours, samples in bags within 48 hours and samples in Vacu-Samplers within four days of collection. Samples were not stable long in 1-L bags because of the high surface-to-volume ratio.

The EO concentration profile was measured by taking samples at one-minute intervals during Test 1 Evacuation 1. This test used 12 wt% EO and 88 wt% CFC-12 as the sterilizing gas. The EO concentration ranged from 400 ppmv to 1,500 ppmv and plateaued between 13 and 20 minutes after beginning evacuation. For 12/88 exhausts, EO concentrations decreased to 80 ppmv during Evacuation 6. During Test 3 Evacuation 1, using 100% EO, the EO concentration ranged from 500 ppmv to 3,000 ppmv. For 100% EO exhausts, EO concentrations decreased to 300 ppmv during the post-sterilization cycle. Exhaust EO concentrations decreased between 20 and 70% between Evacuations 1 and 2 and between 55 and 85% between Evacuations 1 and 3. The EO concentration decreased most rapidly in the 100% EO exhaust.

This report contains conclusions and recommendations based on the field test results, a description of the field test site, the grab sampling and analytical method used, results of the field evaluation, test data, and references used to prepare this report.

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SECTION 1

INTRODUCTION

The Source Branch of the Environmental Protection Agency's (EPA) Environmental Monitoring Systems Laboratory (EMSL) at Research Triangle Park, North Carolina, has a program to develop stationary source test methods of known precision and accuracy for use in determining compliance with EPA standards. While participating in this program, Radian Corporation performed two field evaluations of methods for sampling and analyzing ethylene oxide (EO) in the vent stream from a dilute acid scrubber of a commercial sterilizer. The first test was reported earlier.[1] The second test is reported here. The purpose of the second test was to evaluate the ability of the analytical method for quantitating EO in the scrubber emissions. A second purpose was to determine the stability of the EO in the sample containers, and a third purpose was to measure the EO concentration profile and EO-to-oxygen and EO-to-dichlorodifluoromethane (CFC-12) ratios during the initial sterilizer evacuation.

Because EPA has listed EO as a possible hazardous air pollutant, a standardized analytical method is needed to measure EO levels in a vent stream and for determining control equipment performance. In the first field test, reported earlier,[1] the efficiency of the scrubber was measured by taking samples at the scrubber inlet and outlet. The samples were pumped through heated Teflon® lines directly into gas sampling valves of a gas chromatograph. Scrubber outlet gas flow rates were measured using two orifice plates installed in parallel stack extensions. This first test showed that the technology to measure the vent gas flow rate and acquire samples existed but that additional testing of the analytical method was needed.

For the second test, two recommended changes in the analytical method were made:

- off line analysis was used, and
- the analytical column (5% Fluorcol on Carbopack B) temperature was lowered.

Also, two other analytical columns, 1% and 3% SP-1000 on Carbopack B, were evaluated. Evaluation of the 1% SP-1000 column was proposed in the test plan because this column successfully analyzed samples taken from hospital sterilizers, aerators and drains.[2] The 3% SP-1000 column was evaluated to determine if a higher percentage of liquid phase would improve the column performance.

To evaluate the columns, grab samples were taken in either one-liter Tedlar bags, five-milliliter gas-tight syringes or Vacu-Samplers, and analyzed by gas chromatography with flame ionization detection (GC/FID). The grab samples were also used to determine the stability of the vent gas in the sample containers by analyzing the samples in the field and reanalyzing them in the Radian Perimeter Park (PPK) Laboratory. Also, during the first test, the EO concentration profile and EO-to-oxygen and EO-to-CFC-12 ratios were measured during the initial chamber evacuation. The EO concentration profile and ratios may provide useful information in developing a regulation based on maximum EO concentration emitted normalized to a fixed percent oxygen or CFC-12.

Grab sampling with off-line gas chromatographic analysis was used because this technology provided a large number of samples for evaluating the analytical method and for determining the EO concentration profile of the exhaust stream. The analytical method was tested at the outlet of the control unit, where the vent gas EO concentrations were in the parts per million by volume range and the vent gas was at ambient temperatures.

The purposes of the field evaluation were:

- To evaluate the analytical method using field samples from an operating commercial sterilizer with a dilute acid scrubber;
- To evaluate the stability of the EO in the sample containers as a function of storage time; and
- To determine the EO concentration profile and the EO-to-oxygen and EO-to-CFC-12 ratios during the initial sterilizer evacuation.

Section 2 reports the conclusions and recommendations based on the test results. Section 3 describes the properties of EO and provides a basic description of the sterilization process, the sterilization industry, and previously used sampling and analytical methods for EO in scrubber emissions. Section 4 describes the test site and the sampling and analytical methods used. Section 5 contains the test results. Section 6 lists references. The Appendices contain a detailed description of the method, a description of the Vacu-Samplers, and the test data.

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

The following conclusions were based on the field test results and the laboratory reanalysis of the samples and are applicable to three distinct categories: the analytical column, the sampling container, and the determination of the EO concentration profile.

Analytical Column

All of the tested columns were 1/8-inch (3 millimeter) stainless steel columns packed with a liquid phase coated on 60/80 mesh Carbopack B. The 5% Fluorcol column was 10 feet (3 meters) long; the 3% SP-1000 column was 8 feet long (2.4 m); and the 1% SP-1000 column was 6 feet (1.8 m) long.

Linearity--

Based on a one milliliter injection volume, the FID response using these three columns was linear between 31.25 and 100 vol% for CFC-12. Below 31.25 vol% CFC-12 linearity was not evaluated. Using a one-milliliter injection volume, the FID response when using the 3% SP-1000 column had the largest linear range (0.5 to 100 vol% and 2.281 to 1,254 ppmv) for EO at the percent level and at the part-per-million level. At the part-per-thousand level, the FID response when using the 1% SP-1000 column had the largest linear range (22.81 to 5,016 ppmv) for EO. Therefore, the FID response when using the 1% SP-1000 column provides the best linear range for determining the maximum EO concentration emitted from a dilute-acid hydrolytic control unit.

Efficiency--

Of the three columns evaluated, the 5% Fluorcol column operated at 65°C provided the best efficiency for analyzing percent levels of EO. For part-per-million levels of EO the 1% SP-1000 column was the most efficient. At part-per-thousand levels of EO the 5% Fluorcol and the 1% SP-1000 columns were comparable. The 3% SP-1000 column was clearly the least efficient column at all tested temperatures and concentrations. Therefore, the Fluorcol column provides the best efficiency for percent level EO analyses and the 1% SP-1000 column provides the best efficiency for part-per-million level EO analyses.

Resolution--

The 1% SP-1000 and the 5% Fluorcol columns provided comparable resolution of the EO from the CFC-12 at the percent and parts-per-thousand levels. At column temperatures of 45 and 55°C baseline resolution of EO from CFC-12 was obtained on both columns. Both of these columns provided significantly better resolution of the two compounds than did the 3% SP-1000 column. Therefore, the 3% SP-1000 column is not recommended for analyzing samples of 12/88 gas. Resolution was not evaluated at the parts-per-million level.

Retention Time Stability--

The EO retention times were most stable on the 1% SP-1000 column where they varied from 1.38 to 1.46 minutes (± 6%) at 55°C and were least stable on the 5% Fluorcol column where they varied from 2.21 to 5.43 minutes (± 150%) at 55°C. In general the EO retention times varied least at column temperatures of 65°C. The CFC-12 retention times were most stable on the 3% SP-1000 column where they varied from 1.46 to 1.56 minutes (± 7%) at 65°C and were least stable on the 5% Fluorcol column where they varied from 4.72 to 5.57 minutes (± 18%) at 45°C. Therefore, the Fluorcol column should be used only to analyze for EO at the percent levels in situations where the concentration is not expected to change by more than two orders of magnitude.

Limits of Detection and Quantitation--

The 3% SP-1000 column provided the lowest detection and quantitation limits; therefore, it would be the best column for low-level and trace analyses. The limits of detection and quantitation were comparable for the 1% SP-1000 and 5% Fluorcol columns; therefore, the performance of these two columns may not be distinguished by these criteria.

Sample Container

Evaluations were made of the stability of EO in samples collected in one-liter Tedlar bags, five-milliliter gas-tight syringes, and Vacu-Samplers. After four days in an unpunctured Vacu-Sampler, the EO concentrations in four out of seven duplicate samples (57%) were within ± 8% of the EO concentrations in the original samples. The seven duplicate samples in the unpunctured Vacu-Samplers lost an average of 15% of the EO. Therefore, EO samples in unpunctured Vacu-Samplers remained stable for up to four days, with stability defined as over 50% of the samples being within ± 10% of the original concentrations and the average of all EO concentration changes being less than ± 15 percent.

After four or five days in gas-tight syringes, the EO concentrations in only two out of 12 duplicate samples (17%) were within ± 9% of the EO concentrations in the original samples. The twelve duplicate syringe samples lost an average of 20% of the EO. Therefore, EO samples are not stable in syringes for four or five days.

In leak-free bags the EO in 12/88 exhaust at EO concentrations above 1,000 ppmv varied by an averaged relative difference of + 9% after 48 hours. After five to six days the bags with concentrations of EO greater than 1,000 ppmv showed an average EO loss of 22 percent. In the same time period, bags with concentrations of EO less than 500 ppmv showed an average EO loss of 36 percent. After 17 to 18 days the bags with EO concentrations greater than 1,000 ppmv showed an average EO loss of 41 percent. Therefore, samples of 12/88 exhaust containing EO at concentrations above 1,000 ppmv are stable in Tedlar bags for up to 48 hours.

Leak-free bags containing 100% EO exhaust at EO concentrations above 1,000 ppmv varied by an averaged relative difference of + 3% after four days. After eight days these bags averaged a 20% loss and after twelve days an average of 33% of the EO was lost. In leak-free bags of 100% EO exhaust at concentrations below 500 ppmv an average of approximately 20% of the EO was lost after four days. No additional loss was observed in these bags between Day 4 and Day 12. Therefore, samples of 100% EO exhaust containing EO at concentrations above 1,000 ppmv are stable in Tedlar bags for at least four days.

Since the EO loss in the bags is concentration dependent and more EO is lost at lower concentrations, the EO loss is expected to be caused by adsorption of the EO onto the bag walls. The short sample stability observed in the 1-L bags results from the high surface-to-volume ratio.

Ethylene Oxide Concentration Profile

During the first evacuation of the first test using 12/88 gas, the EO concentration ranged from 400 ppmv to 1,500 ppmv and reached a maximum plateau between 13 and 20 minutes. Concentrations of EO during 12/88 exhausts were approximately 1,200 ppmv during the second evacuation, 600 ppmv during the third evacuation, 300 ppmv during the fourth evacuation, 100 ppmv during the fifth evacuation, and 80 ppmv during the sixth evacuation. During the first evacuation of the third test using 100% EO, the EO concentration ranged from 500 ppmv to 3,000 ppmv. Concentrations of EO during 100% EO exhausts were approximately 600 ppmv during the second exhaust, and 300 ppmv during the third exhaust. Therefore, to measure the maximum EO concentration emitted from a dilute acid hydrolytic scrubber, samples should be acquired between 13 and 20 minutes after the start of the first chamber evacuation when 12/88 is used.

Ratios of the EO-to-CFC-12 concentration were calculated in 12/88 exhaust and a plot of the ratios versus time was similar to a plot of EO concentration versus time due to the high and relatively constant CFC-12 concentration. Oxygen concentrations in the 12/88 exhaust were monitored using Fyrite and indicated that after five minutes the exhaust contained minimal air. Therefore, EO-to-CFC-12 ratios could be established for determining the maximum EO concentration emitted from chambers using 12/88. The EO-to-CFC-12 ratios would only be useful during the first evacuation when the expected CFC-12 concentration is 99.9 percent.

RECOMMENDATIONS

Based on the test results, Radian makes the following seven recommendations. First, a 5% Fluorocol column at 65°C is recommended for measuring percent level EO and CFC-12 concentrations, levels expected at the scrubber inlet or in the exhaust of uncontrolled sterilizers. The FID response when using a 5% Fluorcol column is linear, as indicated by a F-test for linearity, from 0.5 to 50 vol% EO. The 5% Fluorcol column is the most efficient column tested for percent levels of EO, and provides baseline resolution of the CFC-12 and EO at column temperatures of 45 and 55°C and near baseline resolution at 65°C. Also, the retention time instability on the Fluorcol column is less pronounced at percent concentrations.

Second, a 1% SP-1000 column at 45°C is recommended for measuring part-per-million levels of EO, levels expected at the scrubber outlet. The FID response when using the 1% SP-1000 column is linear, as indicated from a F-test for linearity, from 22.81 to 5,016 ppmv EO. The 1% SP-1000 column is the most efficient column tested for parts-per-million levels of EO, and provides baseline resolution of the CFC-12 and EO at 45°C.

Third, it is recommended that a 3% SP-1000 column at 45°C be investigated for quantitating sub-part-per-million levels of EO, levels expected in ambient air at sterilization facilities. The 3% SP-1000 column provided limits of detection and quantitation almost three times lower than the other two tested columns.

Fourth, it is recommended that grab samples collected in gas-tight syringes be analyzed within several hours of collection, samples collected in bags within 48 hours of collection and samples collected in Vacu-Samplers within four days of collection. Syringe samples lost an average of 20% of the EO after four days. In leak-free bags, EO remained stable for at least 48 hours and may be stable for up to four days. After five or six days bag samples lost an average of one-third of the EO. In unpunctured Vacu-Samplers, 57% of the samples were within ± 8% of the expected concentration after four days and the average EO loss for all samples was 15 percent.

Fifth, sampling at least three times between 13 and 20 minutes after the first indication of flow through the stack is recommended for determining maximum EO concentration emitted from a dilute acid hydrolytic scrubber during the first evacuation. The EO concentration in the exhaust increased linearly between five and 13 minutes, plateaued between 13 and 20 minutes, and dropped off after 20 minutes. This time frame corresponds to chamber pressures between 16 and 10 pounds per square inch actual and may need to be adjusted when using a different sterilization program.

Sixth, if the second and following evacuations are monitored, sampling between five and 10 minutes during these evacuations is recommended. During the first five minutes, the gas coming out of the stack is a mixture of gas from the chamber and residual gas from the scrubber so samples taken during this time may be biased high or low.

Seventh, for determining EO-to-CFC-12 ratios during the first evacuation, a 0.1 mL sample volume is recommended for CFC-12 quantitation. A 1 mL sample of percent levels of CFC-12 overloads the analytical column, decreasing the column performance.

SECTION 3

BACKGROUND

PROPERTIES OF ETHYLENE OXIDE

The chemical and physical properties of EO are discussed in References 3, 4 and 5. Only a brief summary is included here. Ethylene oxide, an epoxide, is also called oxirane (the International Union of Pure and Applied Chemistry name).[3] Other synonyms include: dihydrooxirene, dimethylene oxide, 1,2-epoxylethane, oxacyclopropane, oxane, oxidoethane, and α, β-oxidoethane.[3]

It is usually handled as a liquid under pressure. At room temperature and pressure, EO is a gas that has a pungent, irritating, ether-like odor.[3] At 10 degrees Celsius (°C) [50 degrees Fahrenheit (°F)] it condenses to a colorless liquid.[3,4,5] It is completely miscible with water and with organic solvents.[3,4] Ethylene oxide penetrates cardboard, shrink-wrap, paperboard, and product wrappings such as cellophane. Also, EO adsorbs on surfaces and will polymerize or isomerize during storage.

The reactive and volatile properties of EO make it highly flammable and potentially explosive.[3,4] It has a flash point of <-18°C (0.4°F) and is flammable in air at concentrations ranging from 3 to 100 volume percent (vol%).[3,4] It is currently regulated by the Occupational Safety and Health Administration (OSHA) for occupational exposure of 1 part per million (ppm) over 8 hours (hr).[6] EO is a known reproductive hazard[4,7] and a suspected carcinogen.[7] These last considerations, coupled with the fact that there is no upper explosive limit, require that special safety precautions be taken when handling and storing EO.

GENERAL DESCRIPTION OF THE BULK STERILIZATION PROCESS

Detailed descriptions of the sterilization process are contained in References 3 and 8. The bulk sterilization process, used by the majority of the commercial sterilization industry, is described here in general terms. Hospital and medical products to be sterilized are preconditioned with steam in a separate chamber prior to sterilization. An air-tight sterilization chamber is loaded with the preconditioned products. Air inside the chamber is evacuated using steam ejectors to decrease the chamber pressure. If 100 wt% EO is used, the chamber may be purged several times with nitrogen.

Once the desired vacuum level is achieved, the air inside the chamber is humidified to a predetermined value by adding steam. The sterilant is pressurized into the chamber to a predetermined level. The chamber is held at a constant pressure for a specified period, while the products are sterilized by EO. When sterilization is complete, the sterilizer gas mixture is exhausted from the chamber until the chamber pressure is decreased to a predetermined level. Filtered air is introduced into the chamber to flush the sterilant gases from the chamber and product. If 100 wt% EO is used, the chamber is flushed with nitrogen prior to introducing the filtered air. These air washes may be repeated several times. After the last air wash, the door is opened and the chamber unloaded.

The gas in an EO sterilization chamber does not vent at a constant rate for two reasons. First the initial maximum venting rate is determined by the size of the chamber vacuum pump. As the chamber empties, the vacuum is increased, less and less volume of gas is available for removal, and the vent gas flow rate decreases. Second, the evacuation of the chamber is controlled by a solenoid valve that opens and closes at regular intervals to prevent the chamber from evacuating too rapidly and damaging the products. This changing and pulsing flow rate makes measuring the EO mass flow rate in the gas stream more difficult. First, calculating the total flow through the vent is an integration rather than a multiplication process and requires continuous monitoring of the flow. Second, calculating the total emissions requires taking many grab samples over the sampling period or collecting a representative sample using special techniques.

There are two models for the EO concentration profile in the chamber exhaust. In one model the chamber is considered to be a closed system at equilibrium. For this model, the EO concentration in the exhaust gas remains constant and only the vent stream flow rate varies. In the other model, the chamber is considered to be a dynamic system. For this model, both the EO concentration and flow rate of the exhaust gas change with time.

If the sterilization chamber does not contain product, the exhaust stream will be characterized best by the first (static) model because equilibrium will occur quickly between evacuations. However, other than for measuring scrubber efficiency and developing sampling and analytical methods, most chambers are operated with product. Any product present in the chamber will offgas, releasing EO during chamber evacuations and air washes. With a product-containing chamber, the exhaust stream will be characterized best by the second (dynamic) model. From this second, more realistic model, performance factors may be developed and used to study the whole system.

HOSPITAL SUPPLY STERILIZATION INDUSTRY

Throughout the United States and Puerto Rico approximately two hundred facilities use EO for sterilizing heat- or moisture-sensitive products or for fumigating microorganisms and insects.[8,9] Chambers range in volume from less than 2 cubic meters (m^3) [60 cubic feet (ft^3)] to 170 m^3 (6000 ft^3).[9] Most of the chambers in the industry (76%) are charged with a mixture of 12 weight percent (wt%) EO and 88 wt% of dichlorodifluoromethane (CFC-12).[9] The 12/88 gas mixture is used because it is nonflammable. Pure EO is used in 68% of the chambers and mixtures of carbon dioxide and EO are used in 10 percent.[9] Carbon dioxide mixtures are used because the carbon dioxide increases the effectiveness of the EO and prevents it from being flammable. (Some chambers are used with more than one type of sterilant gas.) Some of the 12/88 gas users are converting to 100 wt% EO in anticipation of CFC-12 emission regulations.

The vent streams from 17% of the EO sterilization facilities have some type of control unit, ranging from a neutral-water scrubber to an incinerator.[9] Forty-four percent of the control units chemically hydrolize the EO to ethylene glycol using dilute acid solutions, 3% use catalytic oxidation to convert the EO to carbon dioxide and water, and 19% of the control units are condensation/reclamation systems.[9] Commercial vendors of EO control units include Chemrox Incorporated, Damas Corporation, Donaldson Company (purchasers of Mine Safety Appliances' EO control division), and Croll Reynolds. A segment of the industry (approximately five to 15%) has custom-designed control units. Control units have been tested by the manufacturers and the industry. Ethylene oxide removal efficiencies greater than 99% have been measured.[1,10,11,12,13] Condensation/reclamation and catalytic oxidation systems have lower EO removal efficiencies than the dilute-acid hydrolytic systems.

ETHYLENE OXIDE EMISSION MEASUREMENTS

Several methods are currently being used in the EO sterilization industry to measure the emissions from EO control units.[10,11,12,13] In general, the emissions from an EO control unit are determined on a weight basis over the entire post-sterilization cycle. The post-sterilization cycle includes the initial chamber evacuation and all subsequent air washes. The control units tested and reported include:

- A Chemrox unit in Pennsylvania tested by the manufacturer under contract to the user,[10]
- A Chemrox unit in New York tested by the manufacturer,[11]
- A Damas unit,[12] and
- An MSA unit (now manufactured by Donaldson Company).[13]

The Chemrox unit in Pennsylvania was tested by taking grab samples of the vent gas in Vacu-Samplers every five minutes. The samples were analyzed in the laboratory using standard EO methods. The amount of EO emitted from the control unit was calculated from the concentration of EO in the outlet grab samples, the exhaust temperature, and outlet flow rate of the vent gas. The volumetric gas flow rate was calculated using chamber temperature and pressure data and assuming all components other that EO pass through the scrubber unaffected.

The Chemrox unit in New York was tested using direct semi-continuous sampling at the outlet of the control unit. Samples were directed through heat-traced lines into the gas sampling valve of a gas chromatograph (GC) at one-minute intervals. The weights of EO at the outlet of the control unit were calculated from the concentration of EO measured by the GC and the measured temperature and flow rate of the vent gas.

The Damas unit was tested by taking outlet samples collected on charcoal tubes. A series of outlet samples were collected at a constant sampling rate over five-minute intervals during the entire post-evacuation cycle. The charcoal tubes were desorbed using carbon disulfide and the extracts were analyzed by GC. The weights of EO at the outlet of the control unit were calculated from the measured EO concentrations, vent gas temperature, and vent gas flow rate during each sampling period.

The MSA unit was tested using 10-milliliter (mL) syringes to collect outlet samples at one-minute intervals. Stream flow rates and temperatures were recorded at the time of sample collection. The entire sample in the syringe was injected directly onto a GC column for analysis. The amounts of EO at the outlet of the control system were calculated from the measured EO concentrations and stream flow rates.

SECTION 4

SAMPLING AND ANALYTICAL PROCEDURES

FACILITY DESCRIPTION

The field evaluation was conducted at Burron Medical, a medical supply sterilization facility, located in Allentown, Pennsylvania. The facility has three 1000-ft^3 (28-m^3) sterilizers: one uses a 12/88 (wt%/wt%) EO and CFC-12 gas mixture and two use 100% EO. A 12/88 sterilization cycle typically uses 38 gallons (140 L) of 12/88 gas mixture. On a weight basis, a 12/88 sterilization charge consumes 368 lbs (167 Kg) of gas, of which approximately 44 lbs (20 Kg) are EO. A 100% EO charge typically uses 44 lbs (20 Kg) of gas.

The exhaust from the sterilizers is controlled by a DEOXX™ system. The DEOXX system is a dilute acid scrubber manufactured by Chemrox, which hydrolyzes the EO to ethylene glycol. At the time of the test the scrubber contained dilute sulfuric acid and approximately 10 wt% of ethylene glycol. The control system has reported control efficiencies of 99.99%, based on tests conducted at the facility in April 1986 by the vendor,[10] and 99.94%, based on tests performed in October 1987 by Radian.[1]

Each chamber is equipped with a total recirculating oil-sealed pump. The pumps are equipped with gas/liquid separators that emit the gas to the DEOXX system and recirculate the liquid to the pump inlet. The sterilization cycle is automatically controlled by a programmable microprocessor system. The microprocessor system has the capability to control and record the parameters of the sterilization cycle including chamber temperature, chamber pressure, and elapsed time from the start of the cycle. Because the scrubber is designed to handle the exhaust from two sterilizers venting simultaneously, the tested sterilization cycles were scheduled so that only one sterilizer vented at a time.

Two different sterilization programs plotted in Figures 1 and 2 were used for testing, one for the 12/88 tests (Figure 1) and one for the 100% EO test (Figure 2). Before the start of the tests using 12/88 gas, the chamber was evacuated to 2 pounds per square inch absolute (psia) and then pressurized to 3.1 psia with steam. The steam pressure was maintained for 15 minutes at 3.1 psia and the time period was called a "humidity dwell." At the completion of the humidity dwell the chamber was charged to 23.7 psia with 12/88 gas. The exposure at 23.7 psia was maintained for 15 minutes. The program contained six post-sterilization evacuations, the initial chamber evacuation and pump down and five air in-bleeds and subsequent evacuations, followed by a final air in-bleed. The chamber was evacuated to 2 psia and pressurized with air to 10 psia during each evacuation and air in-bleed cycle. For the sixth air in-bleed the chamber pressure was returned to atmospheric.

Before the start of the test using 100% EO, the chamber was evacuated to 1.5 psia, pressurized to 13 psia with nitrogen, and re-evacuated to 1.5 psia. Then the chamber was pressurized to 2.6 psia with steam. The humidity dwell lasted for 30 minutes. Then the chamber was first pressurized to 6.7 psia with nitrogen, followed by pressurization to 12.1 psia with EO, and capped by pressurizing to 13 psia with nitrogen. The sterilization dwell was maintained for 125 minutes. The program contained five post-sterilization evacuations: the initial chamber pump down, a nitrogen purge and pump down, and three air in-bleeds and subsequent pump downs, followed by a final air in-bleed. For the nitrogen purge the chamber was evacuated to 1.5 psia and pressurized to 13 psia with nitrogen. For the air washes the chamber was evacuated to 1.5 psia and pressurized to 10 psia. For the fourth air in-bleed the chamber pressure was returned to atmospheric.

Three tests were performed, one with product in the chamber using 100% EO and two without product using 12/88 gas. Data from all of these tests were reduced and used to prepare this report. Table 1 summarizes the three tests that were used.

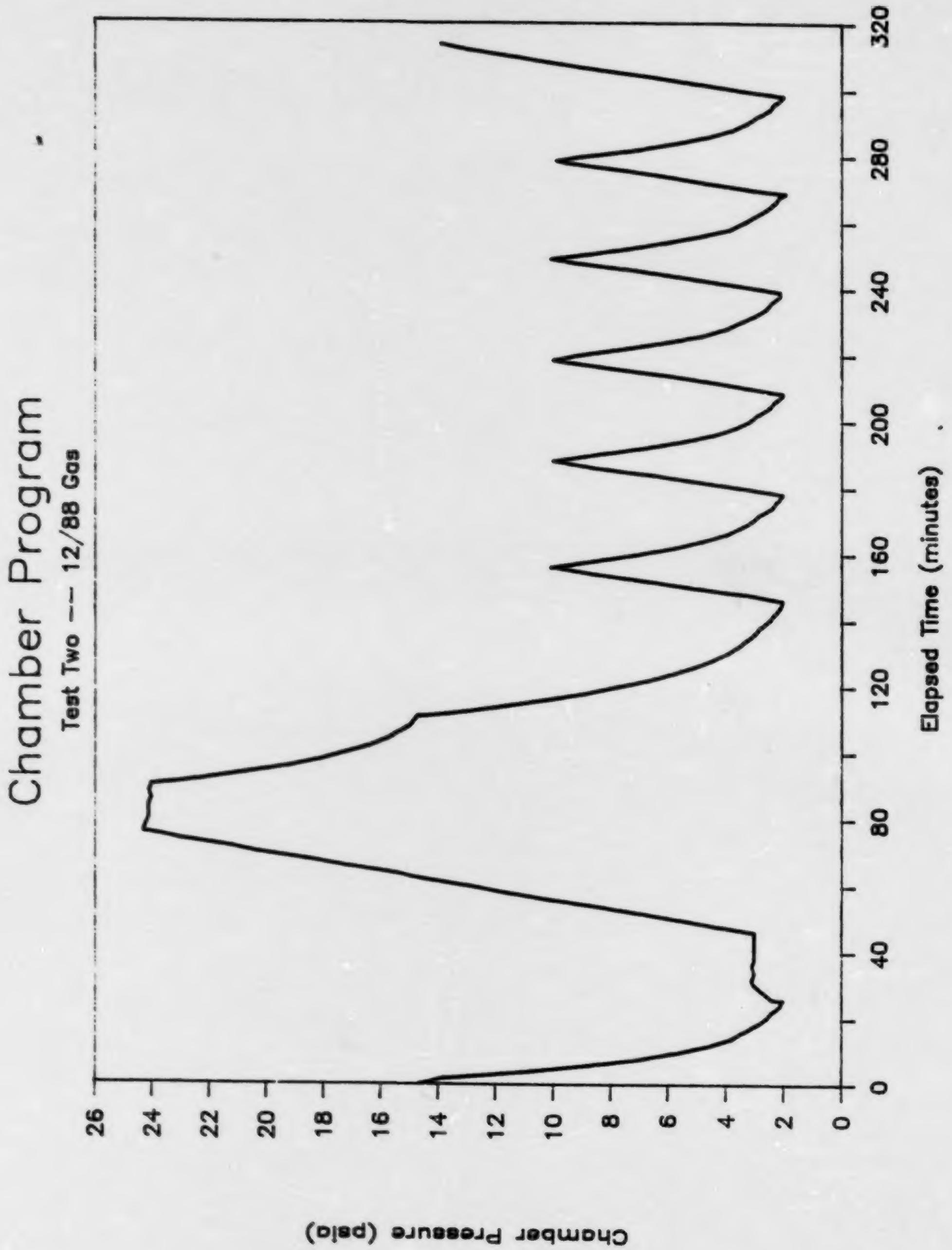

Figure 1. Chamber pressure during tests using 12/88 gas.

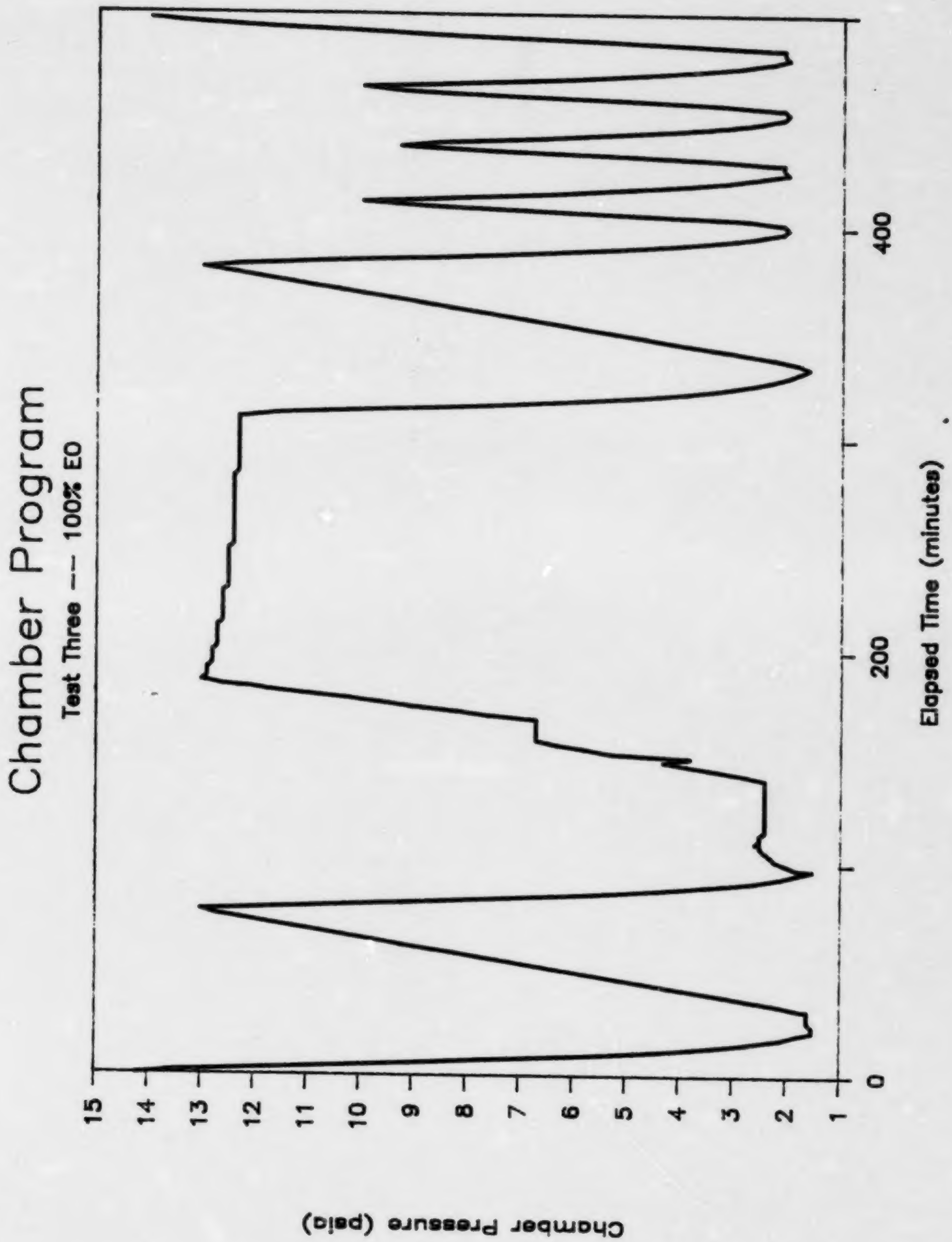

Figure 2. Chamber pressure during tests using 100% ethylene oxide.

TABLE 1. TEST SUMMARY

Test Number	Product Present	Chamber Number	Sterilant Gas Used	Test Date	Test Start Time	Test End Time
1[a]	No	3	12/88	88/06/21	13:03	16:19
2[a]	No	3	12/88	88/06/22	10:00	13:26
3[b]	Yes	1	100% EO	88/06/23	11:45	14:35

[a] The chamber was evacuated to 2 psia before being pressurized with steam to 3.1 psia. Humidity dwell lasted 15 minutes and then the chamber was charged to 23.7 psia with 12/88 gas. Exposure lasted 15 minutes. Post-sterilization chamber pressure cycled between 2 psia and 10 psia.

[b] The chamber was evacuated to 1.5 psia before being filled with nitrogen to 13 psia. After evacuation to 1.5 psia, the chamber was pressurized with steam to 2.6 psia. Humidity dwell lasted 30 minutes and then the chamber was pressurized to 6.7 psia with nitrogen, then to 12.1 psia with EO, and capped at 13 psia with nitrogen. Exposure lasted 125 minutes. The chamber pressure was reduced to 1.5 psia and then pressurized to 13 psia with nitrogen. Post-nitrogen purge chamber pressure cycled between 1.5 psia and 10 psia.

SAMPLING LOCATION

Samples were acquired after the scrubber. At the scrubber outlet the EO concentrations ranged from 80 to 3,000 ppmv, CFC-12 concentrations were at the percent levels, and sample temperatures ranged from 77 to 122°F (25 to 50°C) depending on the ambient temperature at the time of the test.

The stack was modified as shown in Figure 3 to remove exhaust gases from the sampling area and to provide a small amount of back pressure to facilitate sampling and eliminate the possibility of diluting the sample with ambient air. Sample was withdrawn at a point as close to the end of the plant's stack as was feasible.

SAMPLING PROCEDURES

Grab sampling was used for determining EO, CFC-12, and oxygen concentrations. The method used is described in detail in Appendix A.

Ethylene Oxide and Dichlorodifluoromethane Sampling

The EO and CFC-12 sampling equipment is shown in Figure 4. Grab samples were taken using the equipment and procedures described below.

Ethylene Oxide and Dichlorodifluoromethane Sampling Equipment--

Sample was withdrawn into a heated, 1/4-inch (6.4-mm), Teflon® line using a Teflon-lined diaphragm pump. A stainless steel, 1/4-inch (6.4-mm) tee was used after the pump to provide a continuous purge of the vent gas through the sampling system. After the tee, on one branch, the sample stream was routed through tygon tubing and exhausted downwind from the sampling area. There was a pinch clamp on the tygon tubing to block the exhaust line during sample collection. On the other branch of the tee, a stainless steel, 1/4-inch (6.4-mm), toggle-operated shut-off valve was placed prior to the sample container.

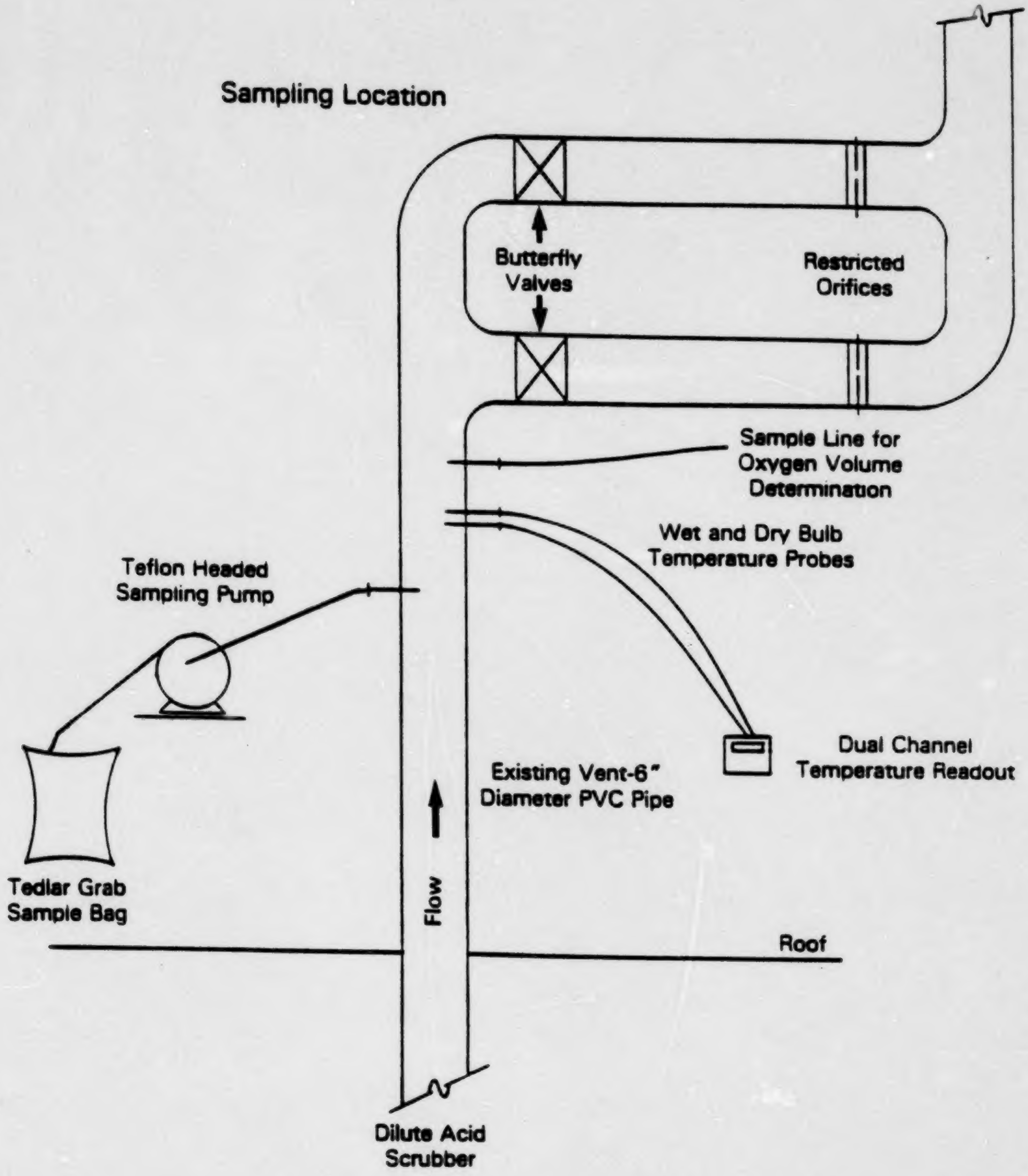
Sampling Location
Butterfly Valves
Restricted Orifices
Sample Line for Oxygen Volume Determination
Wet and Dry Bulb Temperature Probes
Teflon Headed Sampling Pump
Dual Channel Temperature Readout
Existing Vent-6" Diameter PVC Pipe
Tedlar Grab Sample Bag
Flow
Roof
Dilute Acid Scrubber

Figure 3. Stack modification for grab sampling.

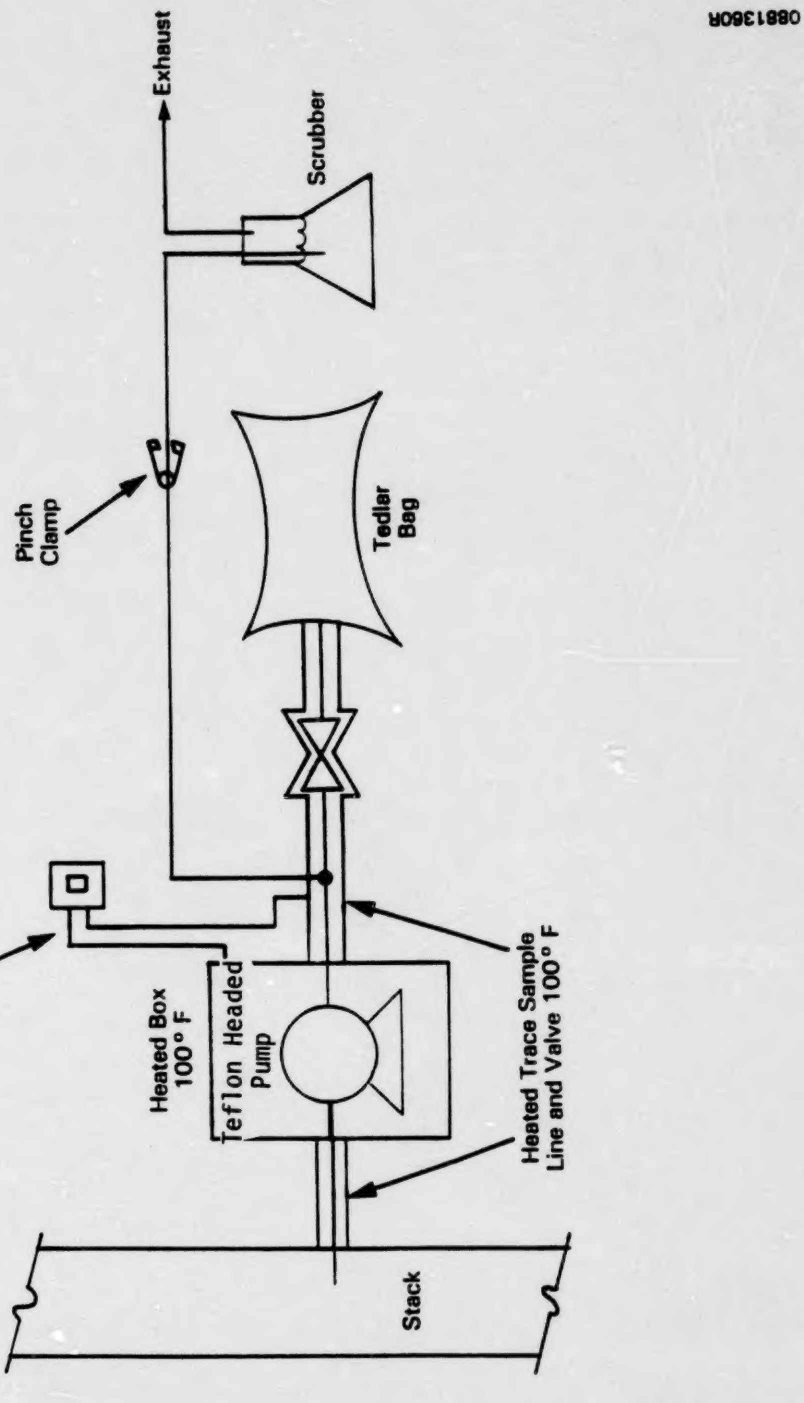

Figure 4. Ethylene oxide sampling equipment.

Ethylene Oxide and Dichlorodifluoromethane Sampling Operation--

The sampling line was continually flushed with sample throughout the test. Prior to attaching the sample container, the line was flushed with sample by closing the pinch clamp on the tygon exhaust line and opening the toggle valve on the sample line. The same procedure was used to fill the sample container.

Table 2 compares the EO and CFC-12 sampling parameters for each evacuation of each test. For the first test, sampling began one minute after the DEOXX scrubber started to exhaust in preparation for the initial chamber evacuation. The test consisted of six evacuations: the initial chamber evacuation and pump down and five air in-bleeds and subsequent evacuations. Sampling stopped eleven minutes after the start of the sixth evacuation. The start time and end time of the evacuations were identified by the chamber control panel.

For the second and third tests, the first sample was acquired five minutes after the start of the first evacuation. After the first sample, bag samples were acquired at six-minute intervals during the first evacuation and at four-minute intervals during the following evacuations.

Oxygen Sampling

The sampling location for oxygen determinations was shown in Figure 3. Grab samples were taken using the materials and procedures described below.

Fyrite Oxygen Indicator--

BACHARACH Fyrite oxygen indicator was used to determine percent levels of oxygen in the sterilizer exhaust at the sampling location. Fyrite indicators use volumetric displacement involving chemical absorption of oxygen from the sample. The reagent used to absorb oxygen was chromous chloride. Accuracy of analysis was ± 0.5 percent.

TABLE 2. COMPARISON OF ETHYLENE OXIDE SAMPLING OPERATION PARAMETERS WITH TEST CONDITIONS

	Test 1		Test 2		Test 3		
	Evacuation		Evacuation		Evacuation		
Parameter	1	2-6	1	2-6	1	2	3-5
Time from start of evacuation to first sample (minutes)	1	5 or 6	5	5	5	5	5
Sampling Interval (minutes)	1	3	6	3 or 4	6	4	3 or 4
Total Samples Acquired							
Bag	24	3	3	3	3	3	3
Syringe	0	0	2	2	1	1	1
Vacu-Sampler	0	0	1	1	2	2	2
Chamber Pressure at start of evac. (psia)	23.7	10	23.7	10	13	13	10
at end of evac. (psia)	2	2	2	2	1.5	1.5	1.5

Sampling Operation--

Percent levels of oxygen were measured when bag samples were taken. The measurements were used to indicate the scrubber dead volume and to determine EO-to-oxygen ratios. Sample was removed from the stack downstream of the EO sampling port. An aspirator bulb was used to pull the sample from the stack.

Stack Temperature

Exit gas temperatures were measured at the scrubber outlet location using a bimetallic temperature probe and a pyrometer. The bimetallic temperature sensor was placed in the duct as shown in Figure 3. Stack temperatures were digitized by a calibrated pyrometer and recorded every time a bag sample was taken.

Moisture Determination

The percent moisture of the stack gas was determined by the wet bulb/dry bulb method. Relative humidity was converted to percent moisture. Wet and dry bulb temperature measurements used to determine relative humidity were recorded every time a bag sample was taken.

ANALYTICAL PROCEDURES

The analytical method used for the measurement of the EO and CFC-12 was gas chromatography with flame ionization detection (GC/FID). The CFC-12 concentration was needed for the determination of the EO-to-CFC-12 ratios. The equipment and procedures used are described below.

Analytical Equipment Description

A dual FID Varian 3400 GC was equipped with a 10 ft (3 m) x 1/8 inch (3 mm) OD stainless steel column containing 5% Fluorcol on 60/80 Carbopack B and a 6 ft (2 m) x 1/8 inch (3 mm) OD stainless steel column containing

1% SP-1000 on 60/80 mesh Carbopack B. On July 9, the 1% SP-1000 column was replaced with an 8 ft (2.4 m) x 1/8 inch (3 mm) OD stainless steel column containing 3% SP-1000 on 60/80 mesh Carbopack B. The FID electrometers were connected to Shimadzu CR1-A integrators.

Operating Conditions

The GC column oven was operated isothermally at 45°, 55° or 65°C on June 21, 22 and 23, respectively. Laboratory reanalyses were performed with an isothermal column temperature of 65°C except on July 9 when the 3% SP-1000 column was used at 55°C. The injector oven was heated to 175°C, and the detector oven to 200°C. Nitrogen carrier gas flow rates were 30 mL/min. The FID support gas flow rates recommended by the GC manufacturer were used.

During the first evacuation of the first test when both EO and CFC-12 were quantitated, the FID electrometers were programmed so that the range would shift from 10^{-10} or 10^{-11} amperes to 10^{-9} amperes.

Analytical Sampling Procedures

One-milliliter samples were injected using a 5-mL gas-tight syringe with an on/off valve.

Gas Chromatograph Calibration

Both channels of the chromatograph were calibrated for EO at the beginning and end of the day. On the first test day, the GC/FID was also calibrated for CFC-12 to determine the EO-to-CFC-12 ratios. At least one standard was analyzed after every twentieth sample. Standards were purchased from Scott Specialty Gases and MG Industries and ranged in EO concentration from less than 5 ppmv to 12.5 vol% and in CFC-12 concentration from 1200 ppmv to 62.5 volume percent. In addition, a lecture bottle containing 99.9% CFC-12 was used to verify response at the upper level of CFC-12 concentration observed in the vent stream. Calibration curves consisted of a minimum of three standards that bracketed the sample concentrations.

SECTION 5

TEST RESULTS

The purpose of this field test was to evaluate the performance of the analytical column. Three columns were evaluated in terms of linearity, efficiency, resolution, retention time stability, and limits of detection and quantitation. A second purpose of the field test was to evalute EO stability in samples collected in Tedlar bags, gas-tight syringes and Vacu-Samplers. A third purpose of the field test was to determine EO-to-oxygen and EO-to-CFC-12 ratios and to measure the EO concentration profile for means of establishing a standard based on maximum EO concentration emitted.

COLUMN EVALUATION

The linearity, efficiency, resolution, retention time stability, and limits of detection and quantitation were evaluated for the following three columns:

- 5% Fluorcol on 60/80 mesh Carbopack B (10 ft x 1/8 inches, stainless steel);
- 1% SP-1000 on 60/80 mesh Carbopack B (6 ft x 1/8 inches, stainless steel); and
- 3% SP-1000 on 60/80 mesh Carbopack B (8 ft x 1/8 inches, stainless steel).

The Fluorcol column and the 1% SP-1000 column were used to analyze samples in the field. All three columns were used to analyze samples in the laboratory. The three columns were evaluated at 45°, 55° and 65°C using a nitrogen carrier gas flowing at 30 mL/min. The results are reported below.

Linearity

The linearity of the EO and CFC-12 response on the FID detector when using the three columns was evaluated using the F-test given in Equation 1:

$$F^* = MSLF/MSPE \qquad \text{(Equation 1)}$$

where:

F^* = the F-test statistic,
MSLF = the lack of fit mean square = SSLF/(c-2),
MSPE = the pure error mean square = SSPE/(n-c),
n = the total number of injections, and
c = the total number of concentrations.

The lack of fit sum of squares (SSLF) is a weighted sum of squares of the deviations between the mean area at each concentration and the fitted area at that concentration. The weights are the number of injections at each concentration. The degree of freedom (c-2) associated with SSLF is two less than the number of concentrations evaluated. Thus the lack of fit mean square (MSLF) provides a variance of the sample points about the fitted line and a smaller MSLF increases the probability that the regression function is linear.

The pure error sum of squares (SSPE) is a sum of squares of the deviations between the area measured for each injection at a concentration and the mean area measured at that concentration. The degree of freedom (n-c) associated with SSPE is the total number of injections less the total number of concentrations evaluated. Thus the MSPE provides an unbiased estimator of the error variance regardless of the nature of the regression function.

The F-statistic, F^*, is compared to a critical F-statistic, $F_{1-\alpha/2,c-2,n-c}$, obtained from tables that provide points for the distribution of F^*. If $F^* > F_{1-\alpha/2,c-2,n-c}$, then the data indicate lack of fit, or non-linear behavior.

The EO concentration may vary over several orders of magnitude depending upon the test situation. Although the FID response for EO is linear from the part-per-million by volume to the volume percent level, a calibration curve prepared over six orders of magnitude must be properly weighted to prevent the volume percent levels from dominating the fitted results. Because proper weighting can complicate calibration curve calculations, the EO concentration range was divided into three fractions with a span limited to 2 or 3 orders of magnitude. No effort was made to determine where non-linearity begins.

Table 3 provides F-test data at the three EO concentration levels and at one CFC-12 concentration level for the FID when using the three columns. At the 0.01 level of significance (99% probability level), FID response for all columns was linear for CFC-12 concentrations between 30 and 100 vol%. For a one milliliter injection volume, CFC-12 concentrations at 30 vol% and higher saturate the FID as indicated by the humped-shaped peaks. Thus, for quantitation of high percent levels of CFC-12, a 0.1 mL sample volume would provide better chromatography. In this study, CFC-12 linearity was not evaluated below 31 vol%.

At a 0.01 level of significance (99% probability level), the FID response using the 3% SP-1000 column had the largest linear range at both the high and low EO levels. Three linear ranges determined for FID response using the 3% SP-1000 column were 2.28 to 1,250 ppmv, 22.8 to 2,510 ppmv, and 0.5 to 100 vol%. The FID response using the 1% SP-1000 column had the largest linear range for intermediate EO concentrations. Linear ranges determined for the FID when using the 1% SP-1000 column were 2.28 to 251 ppmv, 22.8 to 5,020 ppmv and 0.5 to 20 vol%. On the 5% Fluorcol column the FID linear ranges were 2.28 to 502 ppmv, 49 to 2,500 ppmv, and 0.1 to 50 vol%.

Thus, the FID response using the 1% SP-1000 column provided the best linear range for analyses of EO at the outlet of dilute acid hydrolytic scrubbers when 12/88 or 100 wt% EO was used. The FID response using the 5% Fluorcol column provided the best linear range for EO analyses at the scrubber inlet when 12/88 or 100 wt% EO was used. Plots of the calibration curves are provided in Appendix B.

TABLE 3. F-TEST DATA USED TO DETERMINE LINEARITY OF RESPONSE

Concentration	Calculated F-statistic	Degrees of Freedom	Critical[a] F-Statistic	Analytical[b] Column
31 to 100 vol% CFC-12	0.10	2, 10	7.56	5% Fluorcol
2.28 to 502 ppmv EO	1.31	9, 55	2.75	5% Fluorcol
49 to 2,500 ppmv EO	0.67	7, 97	2.82	5% Fluorcol
0.1 to 50 vol% EO	2.13	6, 14	4.46	5% Fluorcol
31 to 100 vol% CFC-12	0.12	2, 14	6.51	1% SP-1000
2.28 to 251 ppmv EO	1.40	4, 6	9.15	1% SP-1000
23 to 5,000 ppmv EO	3.87	5, 7	7.46	1% SP-1000
0.5 to 20 vol% EO	9.81	3, 5	12.06	1% SP-1000
31 to 100 vol% CFC-12	1.73	2, 24	5.61	3% SP-1000
2.28 to 1,250 ppmv EO	0.02	5, 50	3.41	3% SP-1000
23 to 2,500 ppmv EO	2.82	4, 26	4.14	3% SP-1000
0.5 to 100 vol% EO	1.97	5, 34	3.61	3% SP-1000

[a] F-statistics reported at 0.01 level of significance (99% probability level).

[b] All columns were made of 3 mm OD stainless steel and contained the liquid phase coated on Carbopack B (60/80 mesh). All measurements were made using a nitrogen carrier gas flowing at 30 mL/min. Samples were injected via syringe.

Efficiency

Column efficiency in terms of the height-equivalent-to-a-theoretical plate (HETP) was evaluated by injecting one milliliter of samples containing three different EO levels:

- 12.5 vol%,
- 2,508 ppmv, and
- 98.05 ppmv.

The EO retention time and full width at half height were used to calculate the plate number from Equation 2:

$$N = 5.54 \, (t_r/W_{0.5})^2 \qquad \text{(Equation 2)}$$

where:

N = the number of plates,
t_r = the retention time of EO in seconds, and
$W_{0.5}$ = the full width at half height of the EO peak in seconds.

The full width at half height was either obtained directly from the integrator or was measured mechanically by running the chart paper at 50 cm/min. To obtain the HETP, the column length in millimeters was divided by the number of plates. The columns were evaluated at three temperatures and the HETP results are shown in Tables 4 through 6. Smaller HETP values indicate greater efficiency. Values are reported as means with 95% confidence intervals.

For the 12.5 vol% EO sample, the most efficient column was the 5% Fluorcol at 65°C. Surprisingly, for the percent level sample, the efficiency of the columns decreased with decreasing column temperature. For the 2,508 ppmv EO sample, the most efficient columns were the 1% SP-1000 and the 5% Fluorcol operated at 45°C. At this concentration level, the column efficiencies increased with decreasing temperature. For the 98.05 ppmv EO sample, the most efficient column (1% SP-1000) was equally efficient at all three tested temperatures.

TABLE 4. COLUMN EFFICIENCY AT 45°C

Column I.D.[a]	HETP (mm)[b,c] 12.5%	HETP (mm)[b,c] 2.508 ppmv	HETP (mm)[b,c] 98.05 ppmv
1% SP-1000 (1.8 m)	1.58 ± 0.10	1.10 ± 0.05	0.933 ± 0.057
3% SP-1000 (2.4 m)	2.62 ± 0.17	1.94 ± 0.03	1.88 ± 0.07
5% Fluorcol (3.0 m)	1.44 ± 0.05	1.12 ± 0.02	2.16 ± 0.12

[a] All columns were made of 3 mm OD stainless steel and contained the liquid phase coated on Carbopack B (60/80 mesh). All measurements were made using a nitrogen carrier gas flowing at 30 mL/min. A 1-mL sample was injected via syringe. Full widths at half height were measured mechanically for the 3% SP-1000 column, and at the 12.5% EO level at 45°C for the other two columns and at 65°C for the 1% SP-1000 column. All other full widths at half height were measured electronically.

[b] $\mathrm{HETP} = 0.18 \times L \times (W_{0.5}/t_r)^2$

HETP = Height Equivalent to a Theoretical Plate, mm
L = column length, mm
t_r = retention time, EO peak, seconds
$W_{0.5}$ = full-width at half-height, EO peak, seconds

[c] Data are reported as means with 95% confidence intervals

TABLE 5. COLUMN EFFICIENCY AT 55°C

Column I.D.[a]	HETP (mm)[b,c]		
	12.5%	2.508 ppmv	98.05 ppmv
1% SP-1000 (1.8 m)	0.999 ± 0.011	1.22 ± 0.11	0.888 ± 0.069
3% SP-1000 (2.4 m)	3.87 ± 0.12	2.08 ± 0.18	2.26 ± 0.02
5% Fluorcol (3.0 m)	0.883 ± 0.013	1.33 ± 0.03	1.71 ± 0.04

[a] All columns were made of 3 mm OD stainless steel and contained the liquid phase coated on Carbopack B (60/80 mesh). All measurements were made using a nitrogen carrier gas flowing at 30 mL/min. A 1-mL sample was injected via syringe. Full widths at half height were measured mechanically for the 3% SP-1000 column, and at the 12.5% EO level at 45°C for the other two columns and at 65°C for the 1% SP-1000 column. All other full widths at half height were measured electronically.

[b] $HETP = 0.18 \times L \times (w_{0.5}/t_r)^2$

HETP = height equivalent to a theoretical plate, mm
L = column length, mm
t_r = retention time, EO peak, seconds
$W_{0.5}$ = full-width at half-height, EO peak, seconds

[c] Data are reported as means with 95% confidence intervals.

TABLE 6. COLUMN EFFICIENCY AT 65°C

Column I.D.[a]	HETP (mm)[b,c]		
	12.5%	2.508 ppmv	98.05 ppmv
1% SP-1000 (1.8 m)	0.994 ± 0.092	1.69 ± 0.09	0.948 ± 0.093
3% SP-1000 (2.4 m)	2.30 ± 0.02	1.92 ± 0.02	2.02 ± 0.15
5% Fluorcol (3.0 m)	0.792 ± 0.004	1.78 ± 0.05	1.72 ± 0.05

[a] All columns were made of 3 mm OD stainless steel and contained the liquid phase coated on Carbopack B (60/80 mesh). All measurements were made using a nitrogen carrier gas flowing at 30 mL/min. A 1-mL sample was injected via syringe. Full widths at half height were measured mechanically for the 3% SP-1000 column, and at the 12.5% EO level at 45°C for the other two columns and at 65°C for the 1% SP-1000 column. All other full widths at half height were measured electronically.

[b] $\mathrm{HETP} = 0.18 \times L \times (W_{0.5}/t_r)^2$

HETP = height equivalent to a theoretical plate, mm
L = column length, mm
t_r = retention time, EO peak, seconds
$W_{0.5}$ = full-width at half-height, EO peak, seconds

[c] Data are reported as means with 95% confidence intervals

Thus, the 5% Fluorcol column at 65°C was most efficient for percent level EO analyses and the 1% SP-1000 column was most efficient for part-per-million level EO analyses. Both columns at 45°C were comparable in efficiency for part-per-thousand level analyses of EO. The 3% SP-1000 column was clearly the least efficient column at all tested temperatures and concentrations.

Resolution

The column resolution of EO from CFC-12 was evaluated by injecting one milliliter samples at two concentration levels:

- 12.5 vol% EO and 62.5 vol% CFC-12, and
- 2,508 ppmv EO and 6,022 ppmv CFC-12.

The EO and CFC-12 retention times and full widths at the baseline were used to calculate the resolution from Equation 3:

$$R = [2 \times (t_r - t_{r'})]/(W_b + W_{b'}) \qquad \text{(Equation 3)}$$

where:

R = the column resolution,
t_r = the retention time of EO in seconds,
$t_{r'}$ = the retention time of CFC-12 in seconds,
W_b = the full width at the baseline for the EO peak in seconds, and
$W_{b'}$ = the full width at the baseline for the CFC-12 peak in seconds.

The full width at the baseline was obtained mechanically using a chart speed of either 20 or 50 cm/min. The columns were tested at three temperatures and the results are given in Tables 7 and 8. The higher the column resolution, the better the column separates the two peaks. Baseline resolution is given by a column resolution of 1.5 or greater. Values are reported as means with 95% confidence intervals. Examples of the chromatograms are provided in Appendix C.

TABLE 7. COLUMN RESOLUTION FOR VOLUME PERCENT CONCENTRATIONS

Column I.D.[a]	Resolution[b,c] 12.5 vol% Ethylene Oxide and 62.5 vol% Chlorofluorocarbon 12		
	Column @ 45°C	Column @ 55°C	Column @ 65°C
1% SP-1000 (1.8 m)	2.28 ± 0.32	1.70 ± 0.01	0.90 ± 0.21
3% SP-1000 (2.4 m)	1.24 ± 0.18	1.06 ± 0.14	0.64 ± 0.05
5% Fluorcol (3.0 m)	2.04 ± 0.30	1.78 ± 0.24	0.84 ± 0.10

[a] All columns were made of 3 mm OD stainless steel and contained the liquid phase coated on Carbopack B (60/80 mesh). All measurements were made using a nitrogen carrier gas flowing at 30 mL/min. A 1-mL sample was injected via syringe. Full widths at baseline were measured mechanically. Retention times were measured electronically.

[b] $R = [2 \times t_r - t_{r'},)]/(W_b + W_{b'},)$

R = column resolution
t_r = retention time of EO, seconds
$t_{r'}$ = retention time of CFC-12, seconds
W_b = full width at baseline for EO, seconds
$W_{b'}$ = full width at baseline for CFC-12, seconds

[c] Data are reported as means with 95% confidence intervals

TABLE 8. COLUMN RESOLUTION FOR PARTS-PER-THOUSAND CONCENTRATIONS

	Resolution[b] 2.508 ppmv Ethylene Oxide and 6.022 ppmv Chlorofluorocarbon 12		
Column I.D.[a]	Column @ 45°C	Column @ 55°C	Column @ 65°C
1% SP-1000 (1.8 m)	2.72 ± 0.86	1.72 ± 0.61	0.88 ± 0.30
3% SP-1000 (2.4 m)	1.92 ± 1.28	1.58 ± 0.03	1.38 ± 0.13
5% Fluorcol (3.0 m)	2.08 ± 0.25	1.72 ± 0.13	1.40 ± 0.07

[a] All columns were made of 3 mm OD stainless steel and contained the liquid phase coated on Carbopack B (60/80 mesh). All measurements were made using a nitrogen carrier gas flowing at 30 mL/min. A 1-mL sample was injected via syringe. Full widths at baseline were measured mechanically. Retention times were measured electronically.

[b] $R = [2 \times (t_r - t_{r'})]/(W_b + W_{b'})$

R - column resolution
t_r - retention time of EO, seconds
$t_{r'}$ - retention time of CFC-12, seconds
W_b - full width at baseline for EO, seconds
$W_{b'}$ - full width at baseline for CFC-12, seconds

[c] Data are reported as means with 95% confidence intervals.

The highest column resolutions at both concentration levels were obtained at 45°C on the 1% SP-1000 and 5% Fluorcol columns. As expected, the resolution decreased significantly as the column temperature increased.

Thus, the 1% SP-1000 and 5% Fluorcol columns provided comparable (baseline) resolution of the EO from the CFC-12 at the percent and parts-per-thousand levels. Both of these columns provided significantly better resolution of the two compounds than did the 3% SP-1000 column. Resolution was not evaluated at the part-per-million levels.

Retention Time Stability

The retention time stability was evaluated by injecting one milliliter samples at various EO and CFC-12 concentrations. The percent relative difference between the earliest retention time and the latest retention time was calculated and used to compare the column performance at different temperatures and to compare the columns to each other.

Ethylene oxide and CFC-12 retention times at several concentrations and three column temperatures are reported in Tables 9 through 11. Values are reported as means with 95% confidence intervals. Retention times varied by ± 10% or less on the two SP-1000 columns. Figure 5 graphically shows the retention time stability for the 1% SP-1000 column.

On the Fluorcol column the retention times increased with decreasing concentration as depicted in Figure 6. At 45°C the EO retention time shifted from 2.64 to 5.98 minutes (126%) when going from a concentration of 12.5 vol% to 4.454 ppmv. When going from a concentration of 12.5 vol% to 22.81 ppmv the EO retention time shifted from 2.21 to 5.43 minutes (146%) at 55°C and from 1.82 to 2.59 minutes (42%) at 65°C. For changes in CFC-12 concentrations of 62.5 vol% to 6,022 ppmv, the CFC-12 retention time shifted from 4.72 to 5.57 minutes (18%) at 45°C. The CFC-12 retention time shifted from 3.70 to 4.22 minutes (11%) at 55°C and from 2.91 to 3.36 minutes (15%) at 65°C.

Table 9 here

TABLE 9. ETHYLENE OXIDE AND DICHLORODIFLUOROMETHANE RETENTION TIMES ON 1% SP-1000[a]

Ethylene Oxide				Chlorofluorocarbon-12			
Concentration	Retention Times (minutes)[b]			Concentration	Retention Times (minutes)[b]		
(ppmv)	Column @ 45°C	Column @ 55°C	Column @ 65°C	(ppmv)	Column @ 45°C	Column @ 55°C	Column @ 65°C
125,000	1.76 ± 0.05	1.46 ± 0.09	1.15 ± 0.01	625,000	3.16 ± 0.06	2.39 ± 0.18	1.83 ± 0.04
2,508	1.73 ± 0.02	1.41 ± 0.03	1.15 ± 0.02	6,022	3.30 ± 0.03	2.57 ± 0.04	2.02 ± 0.02
502.4	1.71 ± 0.03	NA	NA	1,200	3.22 ± 0.05	NA	NA
98.05	1.77 ± 0.36	1.46 ± 0.03	1.17 ± 0.02				
22.81	1.78 ± 0.08	1.45 ± 0.10	1.19 ± 0.02				
4.451	1.75 ± 0.01	1.38 ± 0.03	NA				

[a] The column was made of 3 mm OD stainless steel and contained the liquid phase coated on Carbopack B (60/80 mesh). All measurements were made using a nitrogen carrier gas flowing at 30 mL/min. A 1-mL sample was injected via syringe. Retention times were measured electronically.

[b] Data are reported as means with 95% confidence intervals.

NA = Not analyzed

TABLE 10. ETHYLENE OXIDE AND DICHLORODIFLUOROMETHANE RETENTION TIMES ON 5% FLUORCOL[a]

Ethylene Oxide				Chlorofluorocarbon-12			
Concentration	Retention Times (minutes)[b]			Concentration	Retention Times (minutes)[b]		
(ppmv)	Column @ 45°C	Column @ 55°C	Column @ 65°C	(ppmv)	Column @ 45°C	Column @ 55°C	Column @ 65°C
125,000	2.64 ± 0.08	2.21 ± 0.01	1.82 ± 0.03	625,000	4.72 ± 0.08	3.70 ± 0.03	2.91 ± 0.03
2,508	2.81 ± 0.05	2.27 ± 0.02	1.89 ± 0.03	6,022	5.57 ± 0.07	4.22 ± 0.11	3.36 ± 0.05
502.4	3.20 ± 0.34	NA	NA	1,200	5.38 ± 0.11	NA	NA
98.05	3.48 ± 0.14	2.79 ± 0.18	2.41 ± 0.15				
22.81	4.69 ± 3.54	5.43 ± 1.45	2.59 ± 0.08				
4.451	5.98 ± 1.55	NA	NA				

[a] The column was made of 3 mm OD stainless steel and contained the liquid phase coated on Carbopack B (60/80 mesh). All measurements were made using a nitrogen carrier gas flowing at 30 mL/min. A 1-mL sample was injected via syringe. Retention times were measured electronically.

[b] Data are reported as means with 95% confidence intervals.

NA = Not analyzed

TABLE 11. ETHYLENE OXIDE AND DICHLORODOFLUOROMETHANE RETENTION TIMES ON 3% SP-1000[a]

Ethylene Oxide				Chlorofluorocarbon-12			
Concentration	Retention Times (minutes)[b]			Concentration	Retention Times (minutes)[b]		
(ppmv)	Column @ 45°C	Column @ 55°C	Column @ 65°C	(ppmv)	Column @ 45°C	Column @ 55°C	Column @ 65°C
125,000	1.49 ± 0.18	1.30 ± 0.18	1.24 ± 0.03	625,000	1.98 ± 0.09	1.68 ± 0.27	1.46 ± 0.03
2,508	1.36 ± 0.27	1.24 ± 0.27	1.21 ± 0.18	6,022	2.08 ± 0.27	1.76 ± 0.18	1.56 ± 0.18
98.05	1.38 ± 0.72	1.26 ± 0.09	1.23 ± 0.05				
22.81	1.36 ± 0.00	1.23 ± 0.18	1.21 ± 0.02				

[a] The column was made of 3 mm OD stainless steel and contained the liquid phase coated on Carbopack B (60/80 mesh). All measurements were made using a nitrogen carrier gas flowing at 30 mL/min. A 1-mL sample was injected via syringe. Retention times were measured electronically.

[b] Data are reported as means with 95% confidence intervals.

NA = Not analyzed

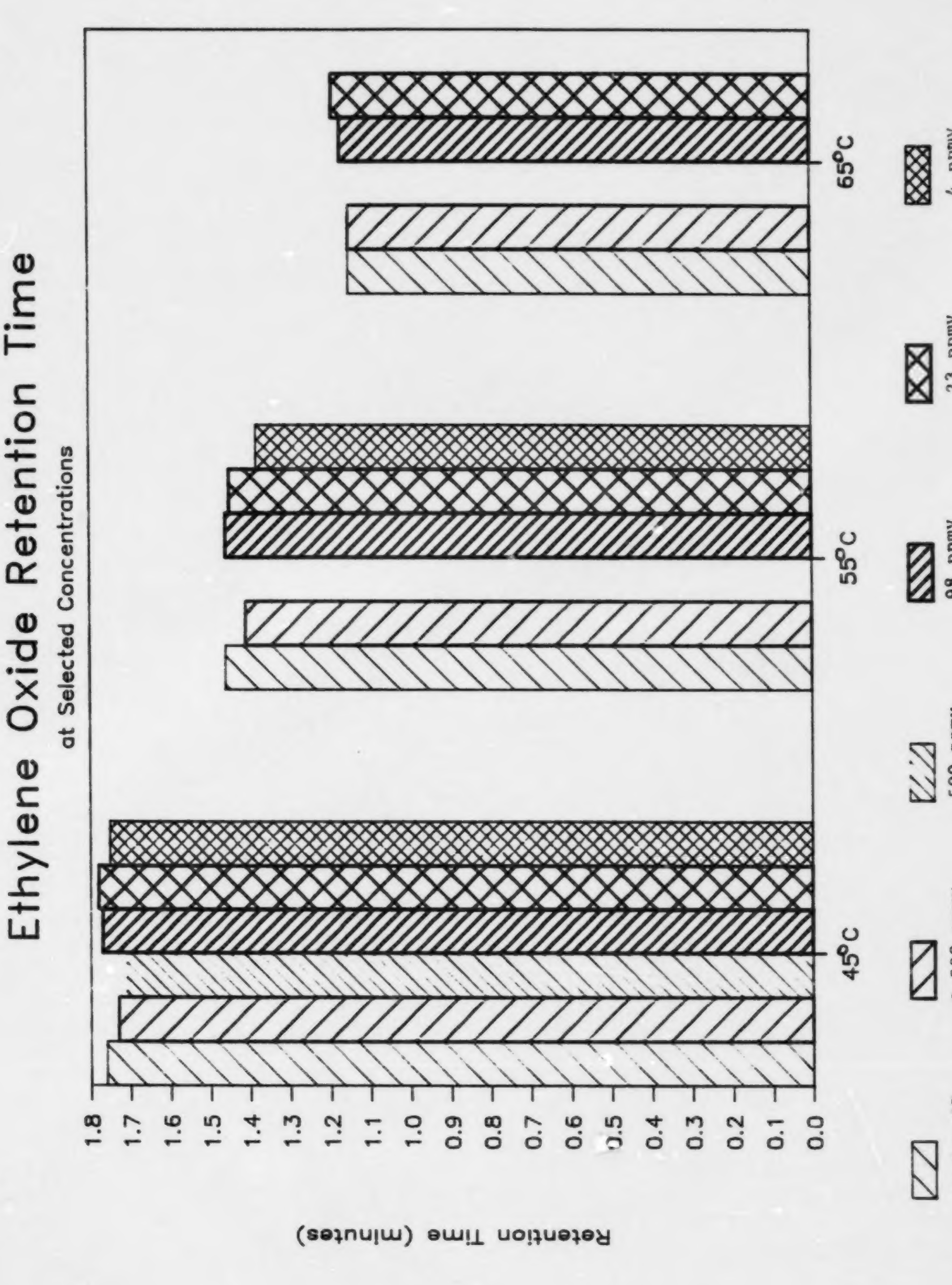

Figure 5. Ethylene oxide retention times on a 1% SP-1000 column.

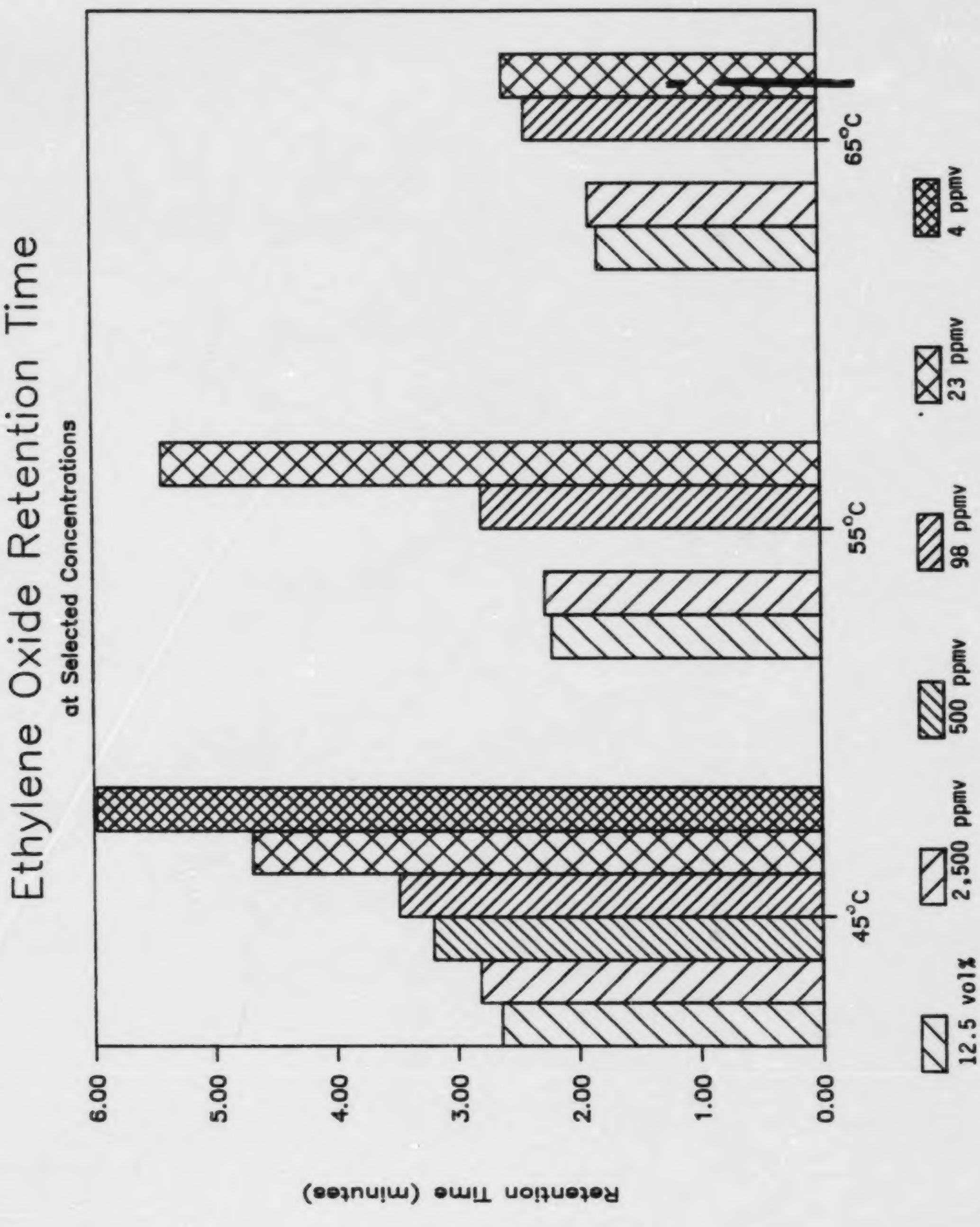

Figure 6. Ethylene oxide retention times on a 5% Fluorcol column.

Thus, the SP-1000 columns were superior for analyzing samples where the expected concentration was unknown or where the concentration was expected to vary by several magnitudes. The Fluorcol column is most useful for analyzing samples with percent level EO concentrations where the retention time shift is minimal. Because of the retention time stability, the 1% SP-1000 column can be used for both scrubber inlet and outlet analyses, whereas the Fluorcol column can not.

Limits of Detection and Quantitation

The limits of detection (LOD) and quantitation (LOQ) were determined using the method reported by Knoll.[14] The columns were evaluated at three temperatures and the results are reported in Tables 12 and 13. Lower LOD and LOQ values indicate columns better suited for trace analyses. Values are reported as means with 95% confidence intervals.

The LOD and LOQ are lowest for the 3% SP-1000 column and are independent of column temperature. For the 1% SP-1000 and 5% Fluorcol columns the LODs and LOQs are comparable and approximately a factor of three greater than for the 3% SP-1000 column.

SAMPLE CONTAINER

The stability of EO was evaluated in three sample containers:

- One-liter Tedlar bags,
- Five-milliliter gas-tight syringes, and
- Vacu-Samplers.

The one-liter Tedlar bags were purchased from NuTech Corporation and had double seams and a twist-valve tube fitting and a septum port for a syringe. Before use they were leak-checked by filling them with inert gas and allowing them to sit overnight under a blanket. Ten percent of the bags were blanked and all of the bags were evacuated prior to sample collection. The bags were not pre-conditioned with sample prior to use.

TABLE 12. LIMIT OF DETECTION[a]

Column I.D.[b]	Concentration (ppmv)[c] Column @ 45°C	Column @ 55°C	Column @ 65°C
1% SP-1000 (1.8 m)	0.17 ± 0.002	0.19 ± 0.004	0.16 ± 0.16
3% SP-1000 (2.4 m)	0.06 ± 0.003	0.07 ± 0.003	0.08 ± 0.002
5% Fluorcol (3.0 m)	0.20 ± 0.03	2.33 ± 0.17	0.14 ± 0.14

[a] Limits of detection and quantitation were determined using the method reported by Joseph E. Knoll in "Estimation of the Limit of Detection in Chromatography," Journal of Chromatographic Science, Volume 23, September, 1985, pages 422-425.

[b] All columns were made of 3 mm OD stainless steel and contained the liquid phase coated on Carbopack B (60/80 mesh). All measurements were made using a nitrogen carrier gas flowing at 30 mL/min.

[c] Data are reported as means with 95% confidence intervals.

TABLE 13. LIMIT OF QUANTITATION[a]

Column I.D.[b]	Concentration (ppmv)[c] Column @ 45°C	 Column @ 55°C	 Column @ 65°C
1% SP-1000 (1.8 m)	0.51 ± 0.007	0.55 ± 0.01	0.46 ± 0.46
3% SP-1000 (2.4 m)	0.18 ± 0.008	0.20 ± 0.008	0.22 ± 0.007
5% Fluorcol (3.0 m)	0.58 ± 0.09	6.79 ± 0.51	0.41 ± 0.41

[a] Limits of detection and quantitation were determined using the method reported by Joseph E. Knoll in "Estimation of the Limit of Detection in Chromatography," Journal of Chromatographic Science, Volume 23, September, 1985, pages 422-425.

[b] All columns were made of 3 mm OD stainless steel and contained the liquid phase coated on Carbopack B (60/80 mesh). All measurements were made using a nitrogen carrier gas flowing at 30 mL/min.

[c] Data are reported as means with 95% confidence intervals.

The five-milliliter gas-tight syringes were Pressure-Lok® Series A-2 with push-button valves. The syringes were used with two-inch tapered needles (0.028 inches OD by 0.008 inches ID). Ten percent of the syringes were filled with inert gas and blanked prior to analysis.

The Vacu-Samplers were purchased from MDA Scientific, Incorporated, and are aerosol-type cans that have been completely evacuated and then back-filled with nitrogen to a partial vacuum of 20 inches of mercury (246.6 mL of nitrogen at standard temperature and pressure). The Vacu-Sampler will collect 123.3 mL of sample at standard temperature and pressure. Additional information on the Vacu-Sampler is contained in Appendix D.

Sample Stability

Duplicate syringe and Vacu-Sampler samples were acquired. One sample was analyzed in the field on the day of acquistion. The other sample was analyzed four or five days later in the Radian PPK laboratory. Stability was determined by calculating percent relative difference using Equation 4:

$$PRD = (C_2 - C_1)/C_1 \times 100 \qquad \text{(Equation 4)}$$

where:

PRD = percent relative difference,
C_2 = measured concentration at PPK laboratory in ppmv, and
C_1 = measured concentration at field site in ppmv.

Stability results for syringes and Vacu-Samplers are listed in Tables 14 and 15, respectively.

Twenty-four of the syringe samples provided reliable stability data. The relative percent differences between duplicate samples analyzed four or five days apart ranged from -40 to +10 and averaged -20 showing that over a four or five day period the EO concentration in the syringes dropped by an average of 20 percent. Previous studies of stability of hydrocarbons in 50-mL syringes

TABLE 14. STABILITY OF ETHYLENE OXIDE IN GAS-TIGHT SYRINGES

Concentration (ppmv) on Analysis Date			Percent[a]	Sterilizing	Analytical
June 22	June 23	June 27	Difference	Gas	Column
1,400	NA	913	-34.8	12/88	1% SP-1000
1,150	NA	1,100	-4.35	12/88	1% SP-1000
1,330	NA	999	-24.9	12/88	1% SP-1000
645	NA	517	-19.8	12/88	1% SP-1000
644	NA	492	-23.6	12/88	1% SP-1000
353	NA	273	-22.7	12/88	1% SP-1000
310	NA	219	-29.4	12/88	1% SP-1000
102	NA	75.7	-25.8	12/88	1% SP-1000
	563[b]	460	-18.3	100% EO	5% Fluorcol
	397[b]	274	-31.0	100% EO	5% Fluorcol
	561[b]	346	-38.3	100% EO	5% Fluorcol
	1,340	1,450	+8.21	100% EO	5% Fluorcol
		average =	-22.1		

[a] Percent Difference is defined as $(C_2 - C_1)/C_1 \times 100$ where C_2 is the concentration on June 27 and C_1 is the concentration on June 22 or 23.

[b] These samples were analyzed on the 1% SP-1000 column and were adjusted for the lower response obtained on the Fluorcol column on June 23 by multiplying the response by 0.77.

NA = Not available

TABLE 15. STABILITY OF ETHYLENE OXIDE IN VACU-SAMPLERS

Concentration (ppmv) on Analysis Date June 23	Concentration (ppmv) on Analysis Date June 27	Percent[a] Difference	Sterilizing Gas	Analytical Column
2,130[b]	2,190	+2.82	100% EO	5% Fluorcol
534[b]	548	+2.62	100% EO	5% Fluorcol
781[b,c]	433	-44.6	100% EO	5% Fluorcol
460	424	-7.83	100% EO	1% SP-1000
778	557	-28.4	100% EO	1% SP-1000
1,480	1,520	+2.70	100% EO	5% Fluorcol
1,460	950	-34.9	100% EO	5% Fluorcol
	average =	-15.4		

[a] Percent Difference is defined as $(C_2 - C_1)/C_1 \times 100$ where C_2 is the concentration on June 27 and C_1 is the concentration on June 23.

[b] These samples were analyzed on the 1% SP-1000 column and were adjusted for the lower response obtained on the Fluorcol column on June 23 by multiplying the response by 0.77.

[c] Sample concentration probably biased high for an unknown reason.

showed that 20% of the hydrocarbons were lost within a four-hour period.[15] The hydrocarbon loss was attributed to diffusion from the syringe.

Only fourteen of the Vacu-Sampler samples were used to provide stability data. The three duplicate Vacu-Sampler samples that had extremely low area counts (below 60 ppmv) were taken at the very end of the evacuation and were suspected of not being true duplicate samples. The relative percent differences between duplicate samples analyzed four days apart ranged from -45 to +3 and averaged -15. Thus, over a four-day period the EO concentration in the Vacu-Samplers dropped by an average of 15 percent. If the one sample suspected of being biased high was eliminated from the data base, the relative difference ranged from -35% to +3% and averaged -10 percent.

Stability results are listed in Tables 16 and 17 for bag samples containing exhaust from 12/88 and 100% EO tests, respectively. For bags containing exhaust from tests using 12/88 gas, the EO remained stable over a 48-hour period. After five to six days, these bags showed an average EO loss of 30 percent. The bags containing EO at concentrations less than 500 ppmv lost 36% and those containing EO at concentrations higher than 1,000 ppmv lost 22 percent. The average EO loss remained at 30% after nine to 10 days and after 13 to 14 days. Only bags with EO concentrations above 1,000 ppmv were analyzed after 17 to 18 days and they showed an average EO loss of 40 percent. Figures 7 and 8 show the variation in measured EO concentration over the 18 day test period for bags containing 12/88 exhaust.

Bags containing 100% EO exhaust at concentrations above 1,000 ppmv remained stable over four days. After eight days these bags averaged a 20% EO loss that increased to 33% after twelve days. Bags with concentrations below 550 ppmv averaged a 20% EO loss that remained unchanged between the fourth and twelfth day. Figures 9 and 10 show the variation in measured EO concentration over the 14-day test period for bags containing 100% EO exhaust.

Since the EO loss in the bags is concentration dependent and more EO is lost at lower concentrations, the EO loss is expected to be caused by adsorption of the EO onto the bag walls. Initially, an equilibrium probably occurs between the bag surface and the gas within the bag accounting for the

TABLE 16. ETHYLENE OXIDE STABILITY IN TEDLAR BAGS CONTAINING 12/88 EXHAUST

	Concentration (ppmv)						
Sample	June 21	June 22	June 23	June 27	July 1	July 5	July 9[a]
Bag #13	1230	1370	1320	995	824	716	733
Bag #20	1340	NA	NA	1010	830	1420	985
Bag #22	1200	1360	1320	956	828	790	760
Bag #25	NA	976	NA	734	562	962	536
Bag #30	NA	374	NA	NA	NA	166	NA
Bag #32	NA	133	96.5	34.2	34.4	NA	NA
Bag #36	NA	28.5	NA	NA	61.8	48.8	65.6
Bag #37	NA	36.7	NA	NA	NA	124	NA
Bag #39	NA	29.8	27.2	1.08	11.9	NA	NA
Bag #41	NA	1170	NA	NA	636	546	538
Bag #44	NA	NA	NA	1120	954	803	810
Bag #45	NA	1240	1380	958	865	690	683
Bag #48	NA	NA	NA	462	370	556	NA
Bag #50	NA	305	340	214	195	173	NA
Bag #55	NA	NA	80.6	57.4	75.2	71.0	NA
Bag #56	NA	NA	101	53.6	70.5	70.6	NA
Bag #57	NA	NA	83.5	51.8	65.8	48.4	NA

[a] Samples analyzed on a 3% SP-1000 column

NA = Not available

TABLE 17. ETHYLENE OXIDE STABILITY IN TEDLAR BAGS CONTAINING 100% EO EXHAUST

	Concentration (ppmv)				
Sample	June 23	June 27	July 1	July 5	July 9[a]
Bag #58	343	NA	270	248	212.8
Bag #60	2,840	2,410	2,030	1,660	1,408.0
Bag #63	544	470	426	430	364.8
Bag #64[b]	342	170	13.7	NA	NA
Bag #65	344	238	258	242	220.8
Bag #66	334	230	268	244	225.6
Bag #67	430	370	322	303	284.0
Bag #68	426	352	307	516	NA
Bag #69	387	282	270	226	NA
Bag #70	1,530	1,760	1,400	1,200	NA
Bag #71	1,440	1,490	1,080	902	NA
Bag #72	1,200	1,270	974	756	NA

[a] Samples analyzed on a 3% SP-1000 column

[b] Bag leaked

NA = Not available

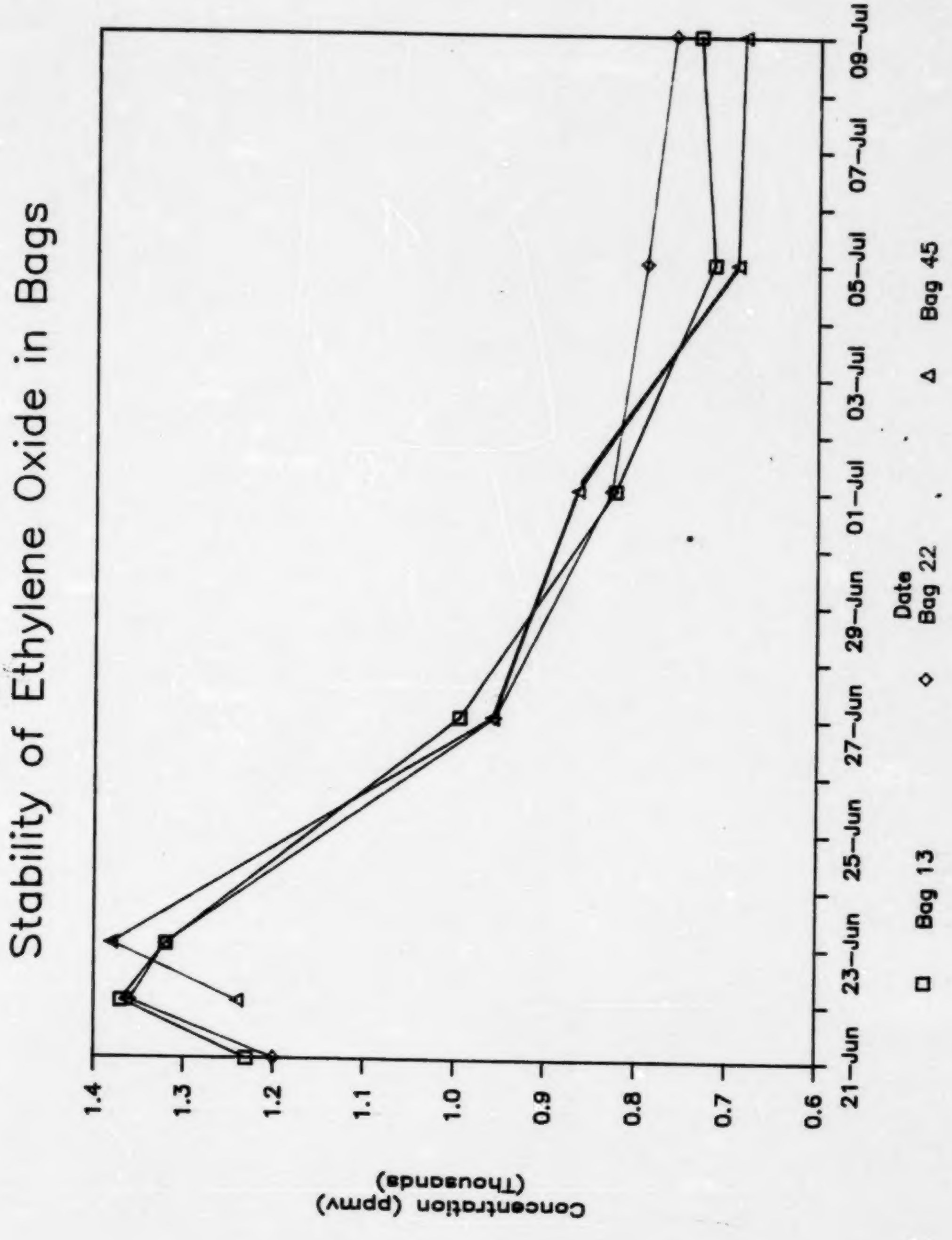

Figure 7. Ethylene oxide stability in 12/88 exhaust at > 1,000 ppmv.

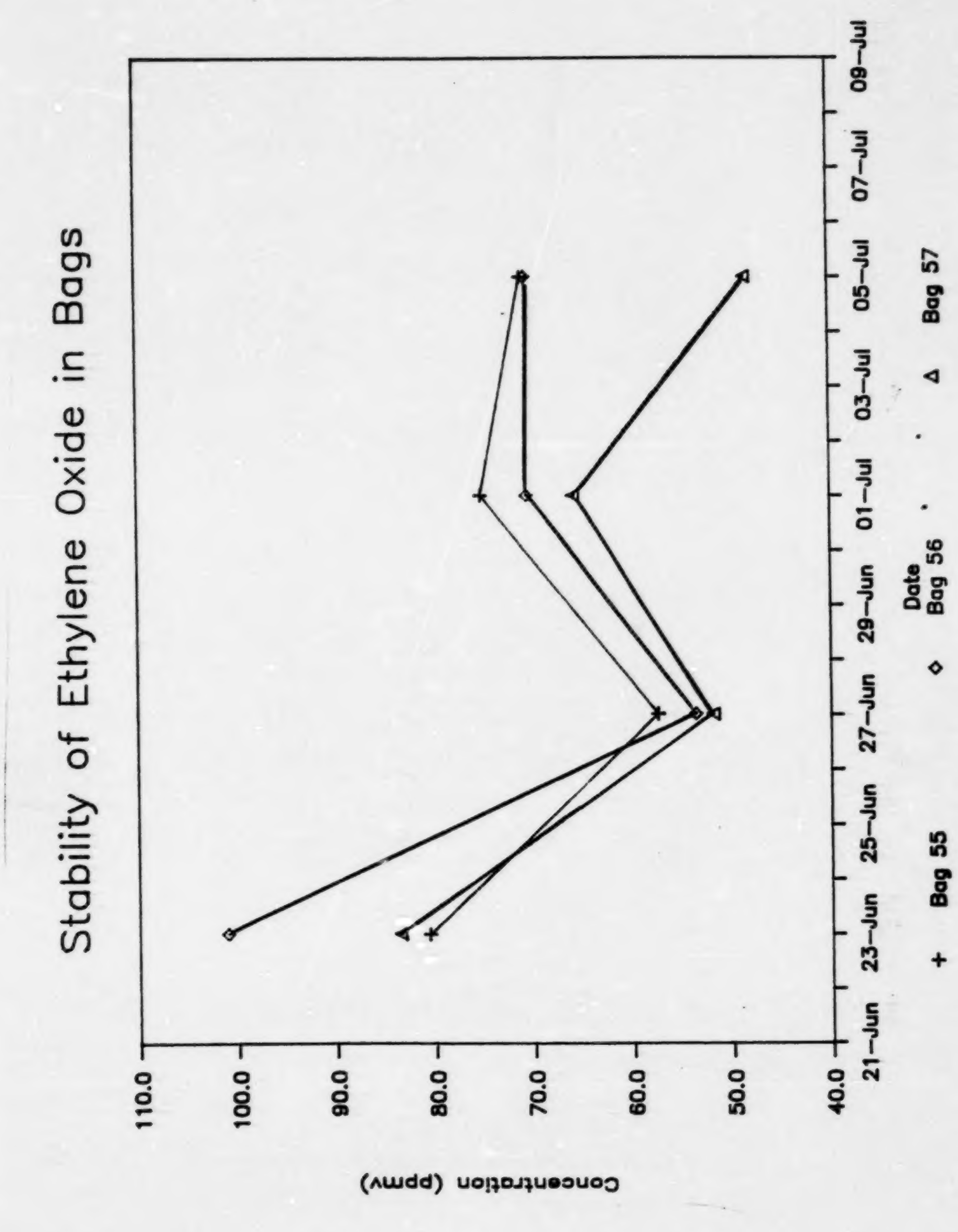

Figure 8. Ethylene oxide stability in 12/88 exhaust at < 100 ppmv.

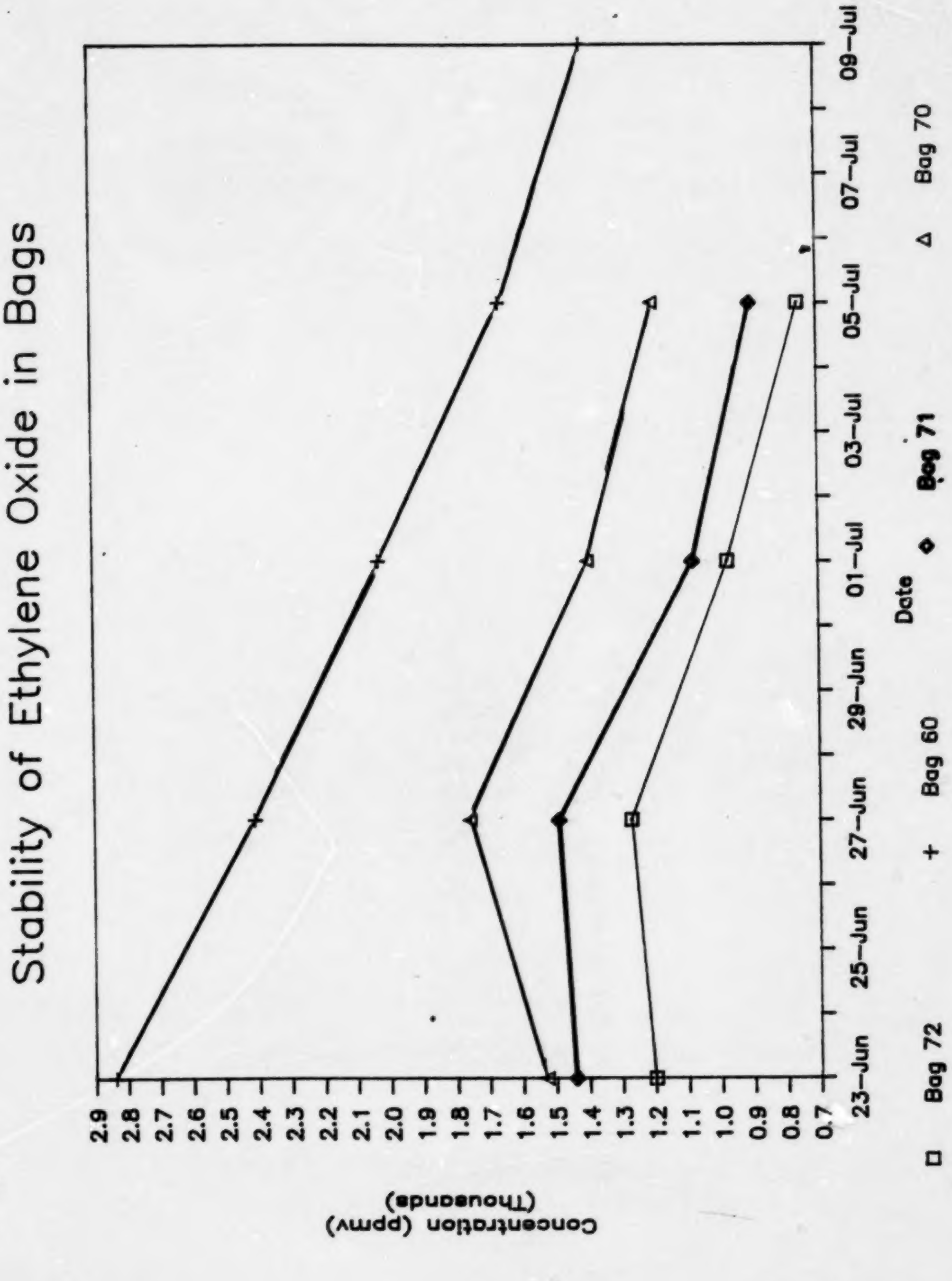

Figure 9. Ethylene oxide stability in 100% EO exhaust at >1,000 ppmv.

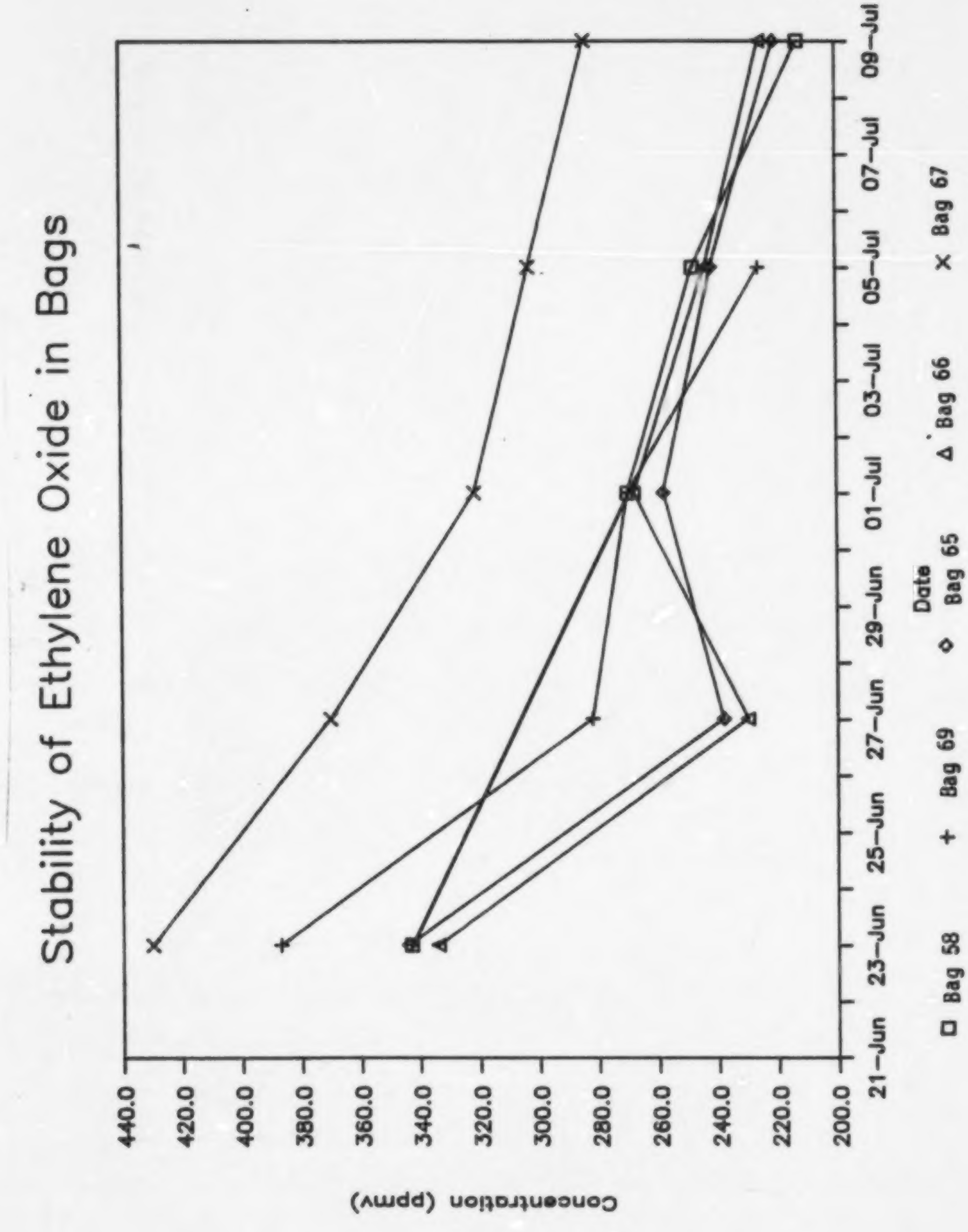

Figure 10. Ethylene oxide stability in 100% EO exhaust at < 500 ppmv.

two- to four-day period of stability observed. As the gas remains in the bag longer, the EO may be penetrating further into the bag wall, accounting for the additional EO loss. Other data[16] shows that improved EO stability can be obtained at surface-to-volume ratios of 3.6×10^{-2} and 2.0×10^{-2} m^2/L. In these bags, 10 ppmv EO samples remained stable for one week.

Sample Container Comparison

During the second and third tests samples were acquired in all three sample containers. Although samples were not acquired simultaneously, they were taken within a two- to three-minute interval so that they would be as identical in composition as possible. The containers were compared by calculating percent relative differences as defined in Equations 5 to 7:

$$PRD_{VB} = (C_V - C_B)/C_B \times 100 \qquad \text{(Equation 5)}$$
$$PRD_{VS} = (C_V - C_S)/C_S \times 100 \qquad \text{(Equation 6)}$$
$$PRD_{SB} = (C_S - C_B)/C_B \times 100 \qquad \text{(Equation 7)}$$

where:

PRD_{VB} = the percent relative difference between EO concentrations measured in Vacu-Samplers and bags,
PRD_{VS} = the percent relative difference between EO concentrations measured in Vacu-Samplers and syringes,
PRD_{SB} = the percent relative difference between EO concentrations measured in syringes and bags,
C_V = the EO concentration measured in the Vacu-Samplers,
C_B = the EO concentration measured in the bag samples, and
C_S = the EO concentration measured in the syringe samples.

The results are given in Table 18.

In general, when looking at the concentrations for comparable samples, the concentrations measured from the three tested containers are usually of similar magnitude. In two cases the syringe samples are obviously low, a

TABLE 18. COMPARISON OF ETHYLENE OXIDE SAMPLE CONTAINERS

Concentration (ppmv) Sample Container			Percent Relative Difference				
Bag	Vacu-Sampler	Syringe	Vacu-Sampler to bag	Vacu-Sampler to syringe	Syringe to bag	Sterilizing Gas	Analytical Column
1,170	Void	1,400	NA	NA	+19.7	12/88	1% SP-1000
1,540	NS	1,150	NA	NA	-25.3	12/88	1% SP-1000
1,430[d]	Void	645[a]	NA	NA	NA	12/88	1% SP-1000
1,240	NS	1,330	NA	NA	+7.26	12/88	1% SP-1000
684[d]	Void	645	NA	NA	-5.70	12/88	1% SP-1000
630[d]	NS	644	NA	NA	+2.22	12/88	1% SP-1000
305	Void	353	NA	NA	+15.7	12/88	1% SP-1000
308[d]	NS	310	NA	NA	+0.649	12/88	1% SP-1000
120	Void	NC	NA	NA	NA	12/88	1% SP-1000
145	NS	175	NA	NA	+20.7	12/88	1% SP-1000
101	Void	102	NA	NA	+0.990	12/88	1% SP-1000
83.5	NS	96.0	NA	NA	+15.0	12/88	1% SP-1000
1,117[a]	2,130[b]	248[a,b]	+90.7	NA	NA	100% EO	5% Fluorcol
2,840	2,602[b]	NS	-8.38	NA	NA	100% EO	5% Fluorcol
560	534[b]	563[b]	-4.64	-5.15	+0.536	100% EO	5% Fluorcol
544	781[b,c]	NS	+43.6	NA	NA	100% EO	5% Fluorcol
344	354[b]	397[b]	+2.91	-10.8	+15.4	100% EO	5% Fluorcol
334	71.5[a,b]	NS	-78.6	NA	NA	100% EO	5% Fluorcol
426	599[b]	561[b]	+40.6	+6.77	+31.7	100% EO	5% Fluorcol
387	465[b]	NS	+20.2	NA	NA	100% EO	5% Fluorcol
1,440	1,480	1,340	+2.78	+10.4	-6.94	100% EO	5% Fluorcol
1,200	1,460	NS	+21.7	NA	NA	100% EO	5% Fluorcol
		Average	+13.1	+0.46	+6.57		

[a] Sample concentration obviously low -- cause unknown.

[b] These samples were analyzed on the 1% SP-1000 column and were adjusted for the lower response obtained on the Fluorcol column on June 23 by multiplying the response by 0.77.

[c] Sample concentration probably biased high for unknown reason.

[d] These samples were analyzed on the 5% Fluorcol column and were adjusted for the higher response obtained on the 1% SP-1000 column on June 22 by dividing the response by 0.82.

NA = Not available

V = Vacu-Sampler pressure was greater than atmospheric.

NS = No sample taken.

NC = Syringe needle was clogged.

conclusion based on analysis of the duplicate syringe sample analyzed for stability purposes and the comparable bag and Vacu-Sampler samples. No known explanation exists for these low syringe sample values. However, possible explanations include a clogged needle, a leaky syringe valve or syringe, and poor sample injection due to a clogged needle, leaky GC septum or operator error. Since these two syringe values were suspected to be low, they were not used in sample container comparisons or in syringe stability determinations.

In one case the Vacu-Sampler data are probably biased high based on the analysis of the duplicate Vacu-Sampler sample and the comparable bag sample. In another case the measured concentration is obviously lower than what was expected based on the comparable bag sample; however, the duplicate Vacu-Sampler sample is also low indicating that the sample may have been taken at the end of the evacuation when flow through the stack was low. In fact, that sample was taken during the last minute of the evacuation when the chamber pressure was 2 psia. For the bag samples, only one sample appears to be obviously and unexplainably lower than expected based on the comparable Vacu-Sampler sample. These data have been included in the sample container comparisons but have been flagged as questionable.

In comparing the Vacu-Samplers with the bags, the percent relative difference for all 10 samples ranged from -80 to +90. The measured EO concentration of samples taken within a two-minute interval averaged 13% higher in Vacu-Samplers than in bags. These data include the questionable Vacu-Sampler data and the questionable bag data. If these data are eliminated from consideration, the percent relative difference ranged from -10 to +40 with the Vacu-Samplers having an EO concentration that averaged 11% higher than the bags.

In comparisons of the Vacu-Samplers to the syringes, the four samples showed relative differences in measured EO concentrations of ± 10 percent. The EO concentration in the Vacu-Samplers averaged 0.5% higher than in the syringes.

The percent relative difference in the EO concentrations measured when comparing bag samples to syringe samples was ± 30 percent. The average relative difference was + 7%, indicating that the EO concentration tended to be higher in the syringe samples. Because the EO concentrations in the bags were also lower than the EO concentrations in the alternate containers, some of the EO was probably lost in the bag samples by adsorption on the bag walls. The EO adsorption losses in the bags were probably increased by not pre-conditioning the bags with sample prior to sample collection.

Another way to compare the sample containers is by the number of valid samples obtained. Out of 34 syringe samples, three samples were lost due to clogged needles, one sample was lost due to a septum leak at the column inlet, and two samples appeared low for an undetermined reason; thus, 17.6% of the syringe samples were invalid. At least 50% of the invalid syringe samples were due to clogged needles, a problem that may be reduced by using sideport needles.

Out of 20 Vacu-Samplers, four samples were inordinately lower than expected and one sample was unexpectedly high; thus, 25% of the samples were questionable. However, eighty percent of the questionable samples were probably due to an inopportune sample time as these samples were taken at the end of the evacuation (chamber pressure less than 2.3 psia) when gas flow through the stack was probably too low to provide an adequate sample.

Only three out of 27 bags (11%) retained for stability analysis had obvious leaks. The three bags were flat after six days. Leaks were not detected in the other 45 bags that were retained but not reanalyzed in the laboratory.

ETHYLENE OXIDE CONCENTRATION PROFILE

Ethylene Oxide Concentration Ranges in the Exhaust Gas

During Test 1 Evacuation 1 using 12/88 gas, grab samples were collected in bags at one-minute intervals. Eighteen samples collected between five and

22 minutes were analyzed. The results are shown in Figure 11. The EO concentration increased from 500 to 1,500 ppmv, indicating that EO volatilization may be more rapid than the decrease in chamber pressure. The desorption rate may be limiting as the maximum concentration plateaued between approximately 13 and 20 minutes. After 20 minutes, the concentration gradually decreased, probably due to decreased volumetric flow.

To summarize the observed EO concentration ranges during each evacuation, the measured EO concentration was 1,200 ppmv during the second 12/88 evacuation, 500 ppmv during the third, 300 ppmv during the fourth, 100 ppmv during the fifth and 80 ppmv during Evacuation 6. When 100% EO was used as the sterilizer gas, higher EO concentrations were measured in the exhaust gas. During Evacuation 1, the EO concentration ranged from 500 to 3,000 ppmv. The measured EO concentration was 500 ppmv during the second 100% EO evacuation, 300 ppmv during the third, 400 ppmv during the fourth, and 1,400 ppmv during the fifth evacuation. The increase in EO concentrations measured during the fourth and fifth evacuations are suspected of being high because Chamber #2 finished its cycle and began exhausting before Test 3 was completed. Since the chambers were all exhausted through one scrubber, attempts were made to prevent two chambers from exhausting simultaneously. This required manual monitoring of the chamber status and was subject to operator error.

Ethylene Oxide Concentration Ratios

During 12/88 tests, both the CFC-12 concentration and oxygen concentration were monitored. Concentration ratios were calculated from these data. Such ratios may be useful in establishing a regulation based on a maximum permissable EO concentration. The Fyrite provides reliable oxygen data for the 12/88 exhausts and can be used to verify whether the plant is diluting the emissions with air. However, if the facility used nitrogen to dilute emissions the dilution would not be detected by this procedure.

The calculated EO-to-CFC-12 ratios ranged from 0.001 to 0.002. A ratio plot as a function of time showed a distribution similar to the concentration profile.

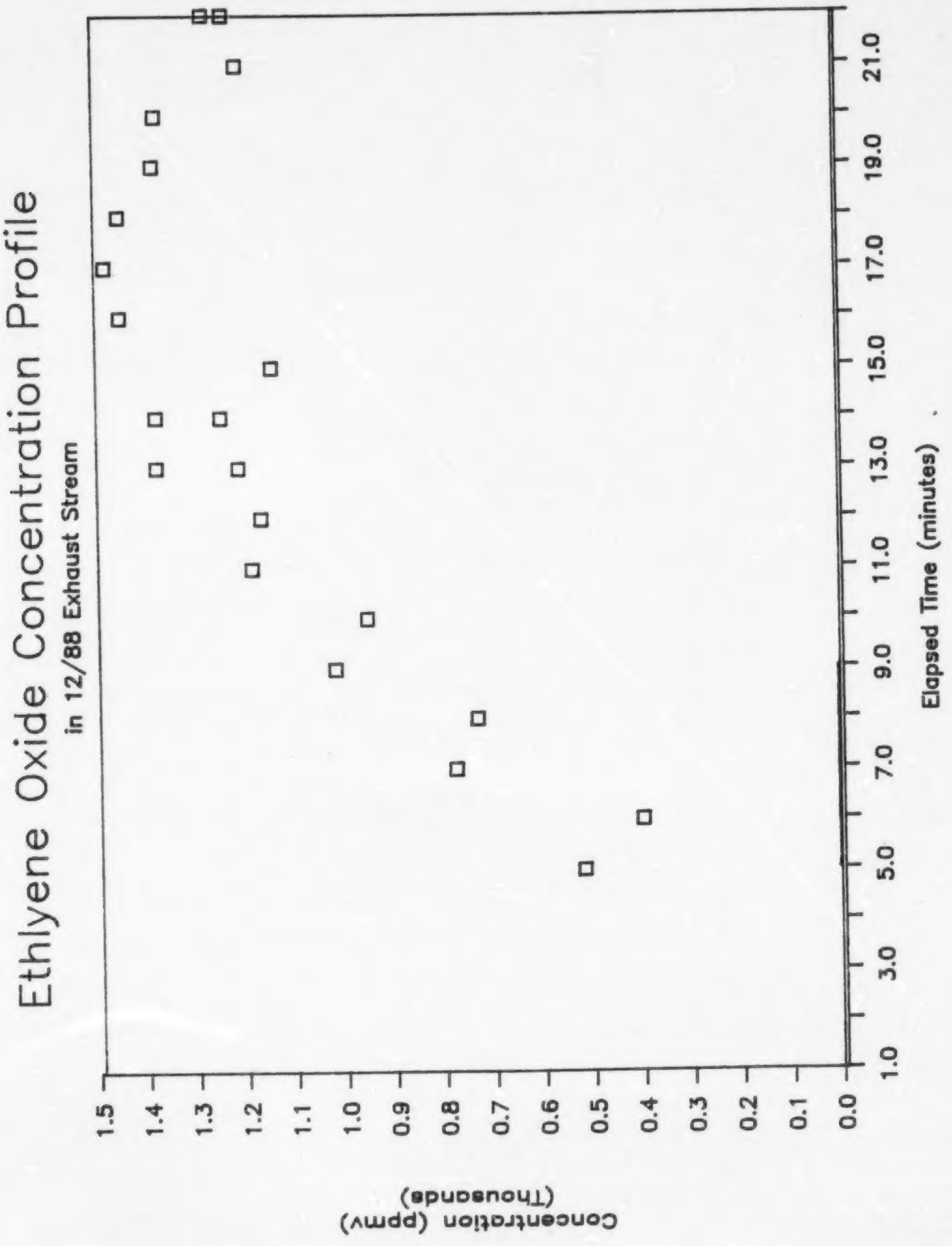

Figure 11. Ethylene oxide concentration profile during the first evacuation.

Unfortunately, Fyrite did not provide reliable oxygen data for 100% EO exhausts; thus, dilution of the exhaust by air cannot be determined. Since no other major components are expected in the 100% EO exhaust, there is no component concentration to which to ratio the EO concentration to indicate that the sample has been diluted.

SECTION 6

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APPENDIX A

GRAB SAMPLING AND ANALYTICAL METHOD DESCRIPTION

APPENDIX A

GRAB SAMPLING AND ANALYTICAL METHOD DESCRIPTION

APPLICABILITY AND PRINCIPLE

Applicability

This method applies to the measurement of the maximum EO concentration normalized to percent oxygen and CFC-12 emitted from sterilization chamber control units. It is applicable to emissions from sterilization control units that use dilute acid hydrolysis to remove the EO. The analytical method is capable of measuring from 0.5 parts per million by volume (ppmv) to 50 volume percent.

Principle

Samples are collected from the sterilization chamber control unit using a 3-second or shorter grab sampling technique and are analyzed off-line by gas chromatography with flame ionization detection (GC/FID). The maximum EO concentration emitted is determined directly from a standard calibration curve and the concentration ratios of EO-to-oxygen and EO-to-CFC-12 are calculated using the measured EO concentrations and the measured oxygen and CFC-12 concentrations.

APPARATUS

The following equipment is required for performing grab sampling with off-line GC/FID analysis.

Heat-traced Teflon® Line

A short heat-traced Teflon line is used to transport the sample from the vent stream to the sample container. The line is heated to a temperature slightly higher than the temperature of the vent gas in order to prevent condensation. The length of line required depends upon the proximity of the sampling container to the sampling port.

Vacuum Pumps

A vacuum pump, capable of pumping 20 liters per minute (L/min) [0.7 cubic feet per minute (cfm)] with a leak-free, Teflon-coated diaphragm, is required to continuously withdraw sample from the sampling port and to pump sample to the sample container.

Acid Scrubber

One 500 mL-Erlenmeyer flask with a two-hole stopper containing dilute sulfuric acid at a pH of 1 is used to remove EO from the sampling stream before venting it to the atmosphere. The acid scrubber may not be required when sampling controlled emissions if the exhaust line is vented in a well-ventilated area void of personnel.

Thermocouples

Two thermocouples are needed to monitor the temperature of the vent stream. One should be wrapped in moist material to measure the wet bulb temperature. The accompanying temperature readout devices should be capable of measuring from ambient temperature up to 200°F (93°C).

Gas Chromatograph with Flame Ionization Detectector(FID)

A GC with a column and FID detector is needed for the off-line analysis of the grab samples. Other detectors (Thermal Conductivity Detector or Photoionization Detector) may be substituted for the FID if they are shown to give equivalent results.

Chromatographic Column

For controlled exhausts, a stainless steel, 6 foot (ft) [1.8 meter (m)] x 1/8 inch [3 millimeter (mm)] outside diameter (OD), column containing 1% SP-100 on 60/80 mesh Carbopack B is used to separate the EO from the CFC-12 and other materials present in the vent gas. For uncontrolled exhausts a stainless steel 10 foot (3m) x 1/8 inch (3mm) OD column containing 5% Fluorcol on 60/80 mesh carbopack B is used. Other columns (Chromosorb 102, Porapak® Q or R, Carbowax 20M) may be used, provided that the precision and accuracy of the standards analysis are not impaired. Information confirming the adequate resolution of the EO peak must be presented. Adequate resolution is defined as an area overlap of not more than 10% of the EO peak by an interferent peak.

Recorder/Integrator

A recorder/integrator is needed to record results.

Regulators

The following regulators are required for the GC support gases and the EO standards.

CGA 580--

One regulator is needed for the nitrogen carrier gas. A second regulator may be required for the standard manifold purge if the carrier gas line cannot be tapped.

CGA 590--

One regulator is needed for the air for the FID.

CGA 350--

Five regulators are needed; one regulator for the hydrogen on the FID and four regulators for the EO and CFC-12 standards. Two regulators with stainless steel diaphragms and Teflon seats and seals are required for the low concentration standards; one should be reserved for standards under 100 ppmv

EO and the other for standards between 100 and 1000 ppmv EO. The other two standard regulators must be stainless steel; one should be reserved for standards between 1000 ppmv and 0.5 vol% and the other for standards above 0.5 volume percent.

Teflon Tubing

Teflon tubing is needed to connect the gas cylinders to the GC, the sampling line to the exhaust area, and for other miscellaneous connections. The diameter and length of the tubing depend upon the requirements of the system used. A recommended amount and diameter of tubing would be 10 to 20 ft (3 to 6 m) of 1/4-inch OD tubing. Each standard regulator should be equipped with 4 to 5 ft (1 to 2 m) of 1/8-inch OD Teflon tubing.

Fittings

An assortment of Swagelock® fittings is desirable to plumb the GC to the gas cylinders and the sample containers to ensure leak-tight fittings. The size and type of fittings needed depend upon the type of tubing used and the type of fittings required by the GC and the cylinder regulators. Some recommendations are:

Caps and Front and Back Ferrules--

Teflon caps and ferrules (1/4-inch) are needed to plumb in the glass flask. Stainless steel caps and ferrules (1/4-inch and 1/8-inch) are used on the ends of the Teflon tubing.

Unions--

Teflon (1/4-inch) and stainless steel (1/4-inch and 1/8-inch) unions are used to connect tubing to impingers, etc.

Reducing Unions--

Stainless steel reducing unions (3/8-inch to 1/4-inch and 1/4-inch to 1/8-inch) are needed to connect cylinders to the GC and the sampling line to the sample containers.

Soap Film Flow Meter

A soap film flow meter is used to measure GC carrier and support gas flow rates. It is also needed to calibrate any rotameters, dry gas meters, and mass flow meters used.

Heated Box or Large Oven

A heated box or large oven is needed for storing the sample containers prior to analysis to prevent water from condensing out of the sample. The box should be capable of being heated up to 40°C (105°F).

Sample Containers

Two dozen sample containers are needed to hold the grab samples. The containers should be clean and evacuated.

Gas-tight Syringe

A 2-mL gas tight syringe with an on-off valve is required for injection of the grab samples into the GC/FID.

Two- or Three-way Radio

A two- or three-way radio is helpful to simplify communications between personnel at the sampling port, in the analytical area, and in the sterilizer control room.

REAGENTS

Unless otherwise indicated, all reagents should conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society where such specifications are available; otherwise, the best available grade should be used.

Nitrogen Gas

A grade of nitrogen which is 99.995 percent pure is required for use as the chromatographic carrier gas and as the system blank. If lower grades of nitrogen are used, purify the gas using hydrocarbon, water, and oxygen traps.

Hydrogen Gas

A grade of hydrogen which is 99.995 percent pure is required as a support gas for the FID. If lower grades of hydrogen are used, purify the gas using a hydrocarbon trap.

Air

A grade of air which is 99.9999 percent pure is required as a support gas for the FID. If lower grades of air are used, purify the gas using a hydrocarbon trap containing activated carbon.

Ethylene Oxide Standard Cylinders

Ethylene oxide and CFC-12 standards prepared in nitrogen which are certified through direct analysis are recommended for system calibration. The following concentrations balanced in nitrogen are suggested:

- 4,000 ppmv EO and 80 vol % CFC-12,
- 1,000 ppmv EO and 20 vol % CFC-12,
- 250 ppmv EO and 5 vol % CFC-12,
- 62.5 ppmv EO and 1.25 vol % CFC-12.

Dichlorodifluoromethane Lecture Bottle

A lecture bottle of 99% CFC-12 is required for calibrating the analytical system for samples containing CFC-12 at concentrations above 80 vol%.

1 N Sulfuric Acid Solution

A scrubbing solution consisting of 1 N H_2SO_4 with a pH between 1 and 2 is required for removing EO from the sampling stream before venting to the atmosphere. To prepare add 30 mL of concentrated sulfuric acid to 1 L of distilled water. Mix well. A minimum of 500 mL of scrubbing solution is needed.

Quality Assurance Audit Samples

A minimum of one gas audit cylinder from the EPA repository, if possible, in the range of the expected EO concentration is required to verify the accuracy of the analytical system and the competence of the analyst.

PROCEDURE

Sampling Considerations

The sampling period begins four to 13 minutes after the start of the initial chamber evacuation and lasts about seven to 16 minutes, ending when the chamber has been completely evacuated or approximately 20 minutes after the start of the evacuation. The sampling line is continuously flushed with sample during the sampling period. The number of grab samples is based on the length of the evacuation interval and expected EO concentration profile.

Flow Rate Estimation--

If flow rate information in desired, molar gas flow rate may be estimated from chamber temperature and pressure provided from the chamber monitor console.

Temperature--

Record the stack temperature using a type-K thermocouple or equivalent device every time a sample is acquired or every two minutes.

Moisture Content--

Determine the moisture content of the vent stream during each evacuation using the wet bulb/dry bulb technique.

Grab Sampling Procedure

In this procedure, sample is continuously withdrawn from the vent stream using a vacuum pump. The gas is channeled into the sample container during a three-second interval over regularly spaced intervals.

Assemble the sampling system as shown in Figure A-1. Leak check the assembly to prevent sample dilution and to protect personnel from fugitive EO emissions.

Place the probe at the centroid of the stack. Start the vacuum pumps and heat the sample transfer lines to prevent condensation.

When evacuation of the chamber begins, wait at least four minutes for the system dead volume to be flushed out. Record the temperature of the vent gas when a sample is taken or at two-minute intervals. Measure oxygen content at one-minute intervals or each time a sample is taken. Take at least one wet bulb reading during each evacuation. The sample data sheet shown in Figure A-2 should be used to record the necessary sample data.

Analytical Procedure

Grab samples are analyzed by GC/FID. Samples collected in gas-tight syringes should be analyzed within several hours of collection. Samples collected in Tedlar bags should be analyzed with 48 hours of collection and samples collected in Vacu-Samplers should be analyzed within four days.

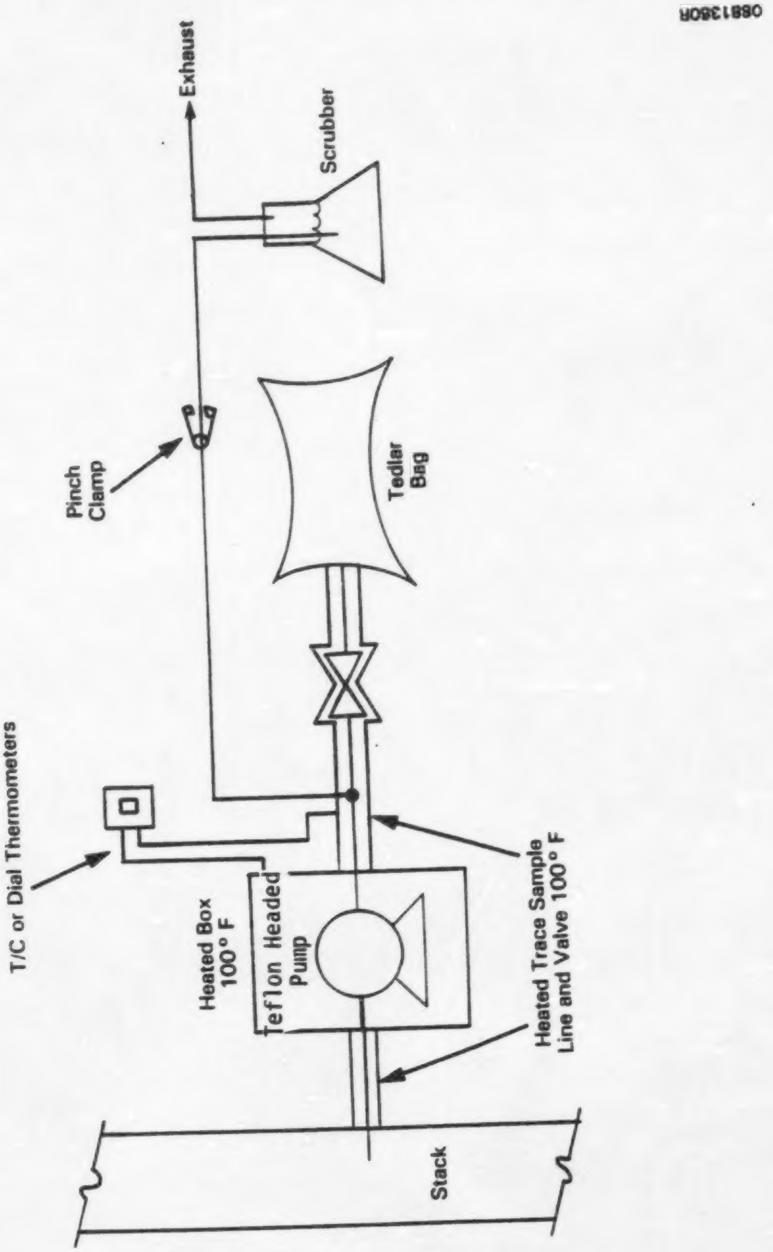
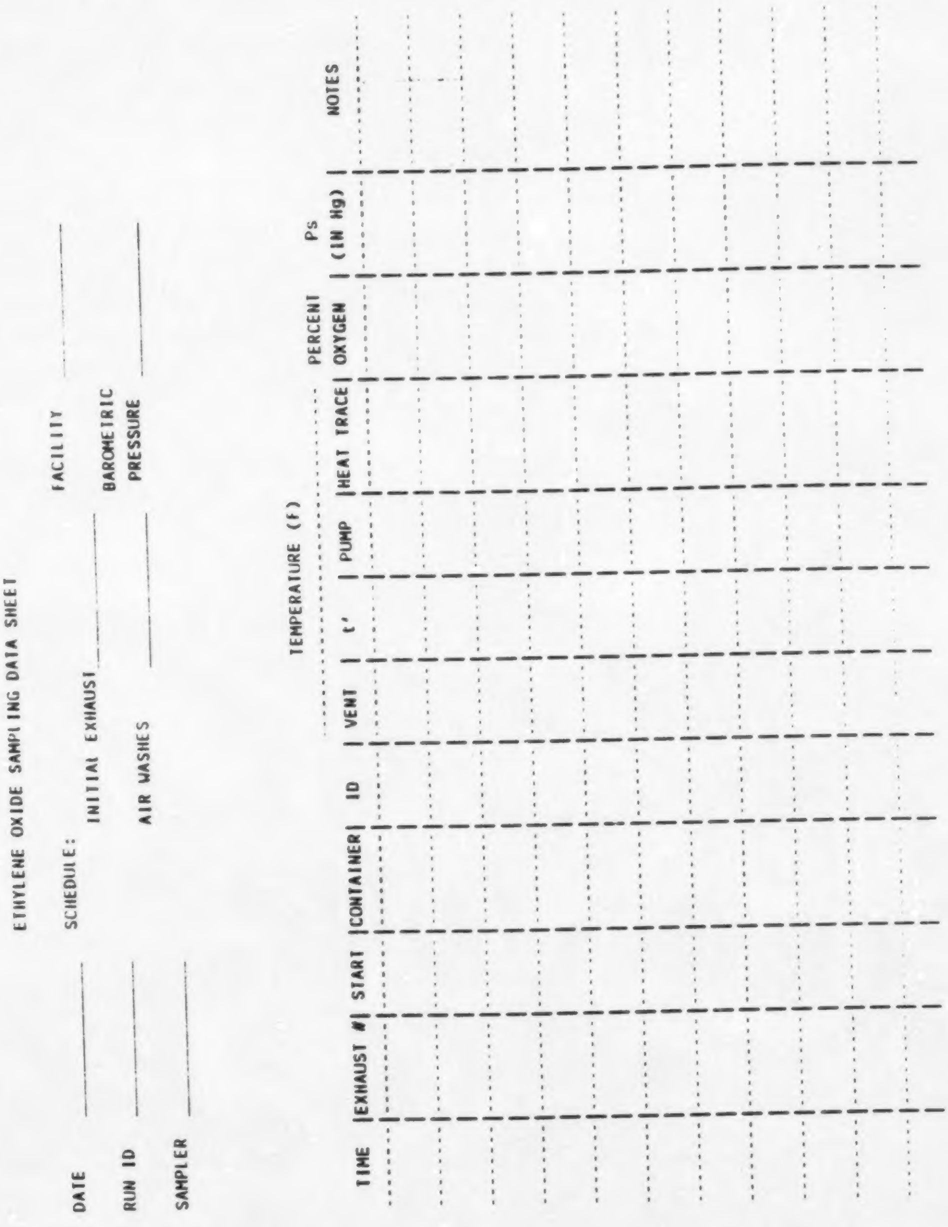


Figure A-1. Grab sampling assembly.



ETHYLENE OXIDE SAMPLING DATA SHEET

DATE ____________

RUN ID ____________

SAMPLER ____________

SCHEDULE:

INITIAL EXHAUST ____________

AIR WASHES ____________

FACILITY ____________

BAROMETRIC PRESSURE ____________

TIME	EXHAUST #	START	CONTAINER	ID	TEMPERATURE (F) VENT	t'	PUMP	HEAT TRACE	PERCENT OXYGEN	Ps (IN Hg)	NOTES

Figure A.2 Sample data sheet.

Gas Chromatographic Conditions--

The chromatographic conditions listed in Table A-1 will resolve EO from CFC-12 and other interferences common to EO control unit vents. It may be necessary to change these conditions to resolve other interferences that are present in samples collected from different EO sources.

Calibration--

Calibrate the GC before the start and at the end of the test using the gas standards. A minimum of four points (four different standard concentrations) are needed to construct a calibration curve. For analysis of samples from a controlled chamber, the GC requires calibration with 60 ppmv to 4,000 ppmv EO standards. For calculation of the EO to CFC-12 concentration ratio, calibrate the GC for CFC-12 using the CFC-12 lecture bottle. Use the same injection method and the same volume of sample for the calibration standards and the samples.

Sample Analysis--

After the GC is calibrated, analyze the samples. After every tenth sample perform a replicate analysis. After every twentieth sample, inject a mid-point standard to verify detector response and stability.

Documentation--

Document each sample and chromatogram by listing the sample location, sample time, sampler initials, vent gas temperature and static pressure, injection volume and time, barometric pressure, room temperature, and analyst initials.

Audit Analysis

Immediately after the preparation of the calibration curve and prior to the sample analyses, perform the analysis audit described in Appendix C, Procedure 2: "Procedure for Field Auditing GC Analysis." 40 CFR, Part 61.

TABLE A-1. GAS CHROMATOGRAPHIC CONDITIONS

Parameter	Recommendation
Column	5% Fluorcol on 60/80 Mesh Carbopack B, 10 ft (3 m) x 1/8 inch (3 mm), stainless steel for percent level analyses; 1% SP-1000 on 60/80 Mesh Carbopack B, 6 ft (1.8 m) x 1/8 inch (3 mm), stainless steel for ppmv level analyses.
Column Temperature	65°C, isothermal for percent level analyses; 45°C, isothermal for ppmv level analyses
Injector Temperature	175°C
Detector Temperature	200°C
Gas Flow Rates	Follow manufacturer's recommendations

CALIBRATION

Probe Temperature Gauge and Thermocouple

Calibrate using ice water and boiling water (ASTM-El #63C or 63F specifications) before the test.

Gas Chromatograph

Calibrate the GC at the start and at the end of each test day using prepared or purchased gas standards. Follow the calibration procedure described earlier. Use the chromatographic data (peak height or area) and standard concentration to prepare a least squares calibration curve.

Certified Standard Cylinders

Verify the certified concentrations of the purchased standard cylinders using an independent standard (one purchased from a second supplier or prepared in the laboratory using pure EO and CFC-12 diluted with nitrogen). Using the independent standard, prepare four-point calibration curves. From the calibration curves, calculate the measured concentrations of the certified standards. If the measured concentrations differ from the certified concentrations by more than ±10%, do not use the standards.

CALCULATIONS

Perform the following calculations, retaining at least one figure more than the required number of significant figures. Round off to the correct number of significant figures after making the final calculation.

Ethylene Oxide Concentration

Determine the EO concentration at each measured point by comparing the area obtained for each sample with the least squares calibration curve obtained as described earlier. Plot EO concentration versus elapsed time.

Reproducibility

Calculate the reproducibility of the analysis by comparing the response of the replicate analysis to the original analysis using the following equation:

$$R = (C_R - C_O)/C_R \times 100 \qquad \text{(Equation A-1)}$$

where:

R = percent difference between replicate analyses,
C_R = the EO concentration in the replicate analysis, and
C_O = the EO concentration in the original analysis.

If more than three replicate analyses are performed per day, report an average R value with a standard deviation.

Inert Gas Concentration

Determine the inert gas concentration at each measured point based on GC data. If no CFC-12 is present in the sample, assume the gas which is not EO is air. Plot the inert gas concentration versus elapsed time.

Moisture Content

Calculate the moisture content (C_a) in the exhaust gas using the following formula:

$$C_a = (VP \times RH)/P_{bar} \qquad \text{(Equation A-2)}$$

where:

C_a = Water vapor in the gas stream, mole fraction
P_{bar} = Barometric pressure, mm (inches) Hg
VP = Saturated vapor pressure of water at stack temperature, mm (inches) Hg
RH = Relative humidity determined by wet bulb/dry bulb method

The relative humidity using the wet bulb/dry bulb method and the saturated vapor pressure of water at stack temperature can be obtained from standard tables.

Normalized Ethylene Oxide Concentration

Calculate the normalized EO concentration by dividing the measured EO concentration in ppmv by the CFC-12 or oxygen concentration in volume percent using the following equation:

$$N_{EO} = C1/C_2 \times 10,000) \qquad \text{(Equation A-3)}$$

where:

N_{EO} = normalized EO concentration,
C_1 = EO concentration, ppmv,
C_2 = CFC-12 or oxygen concentration, vol%, and
10,000 = conversion factor, ppmv/volume percent.

APPENDIX B

CALIBRATION CURVES

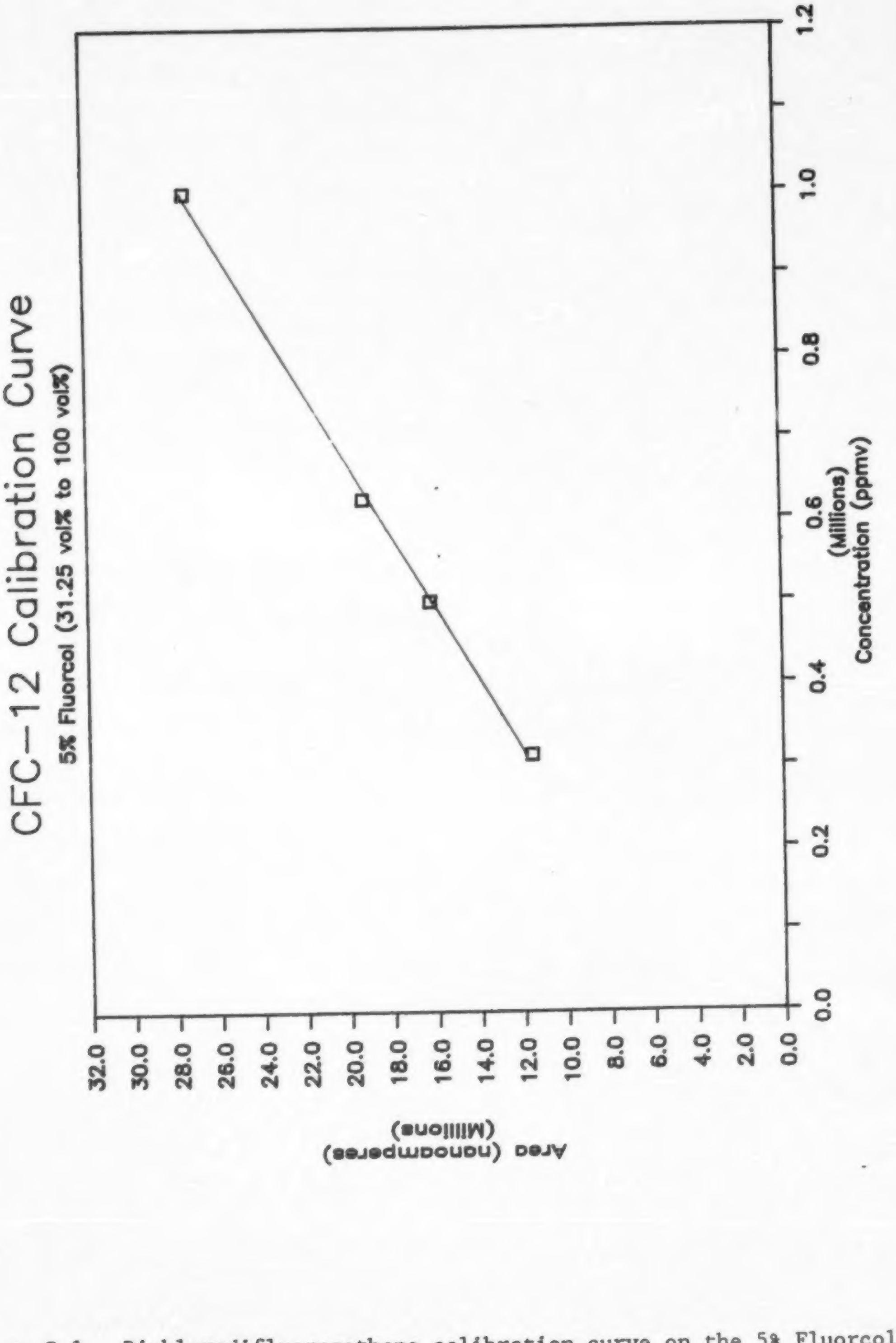


Figure B-1. Dichlorodifluoromethane calibration curve on the 5% Fluorcol column.

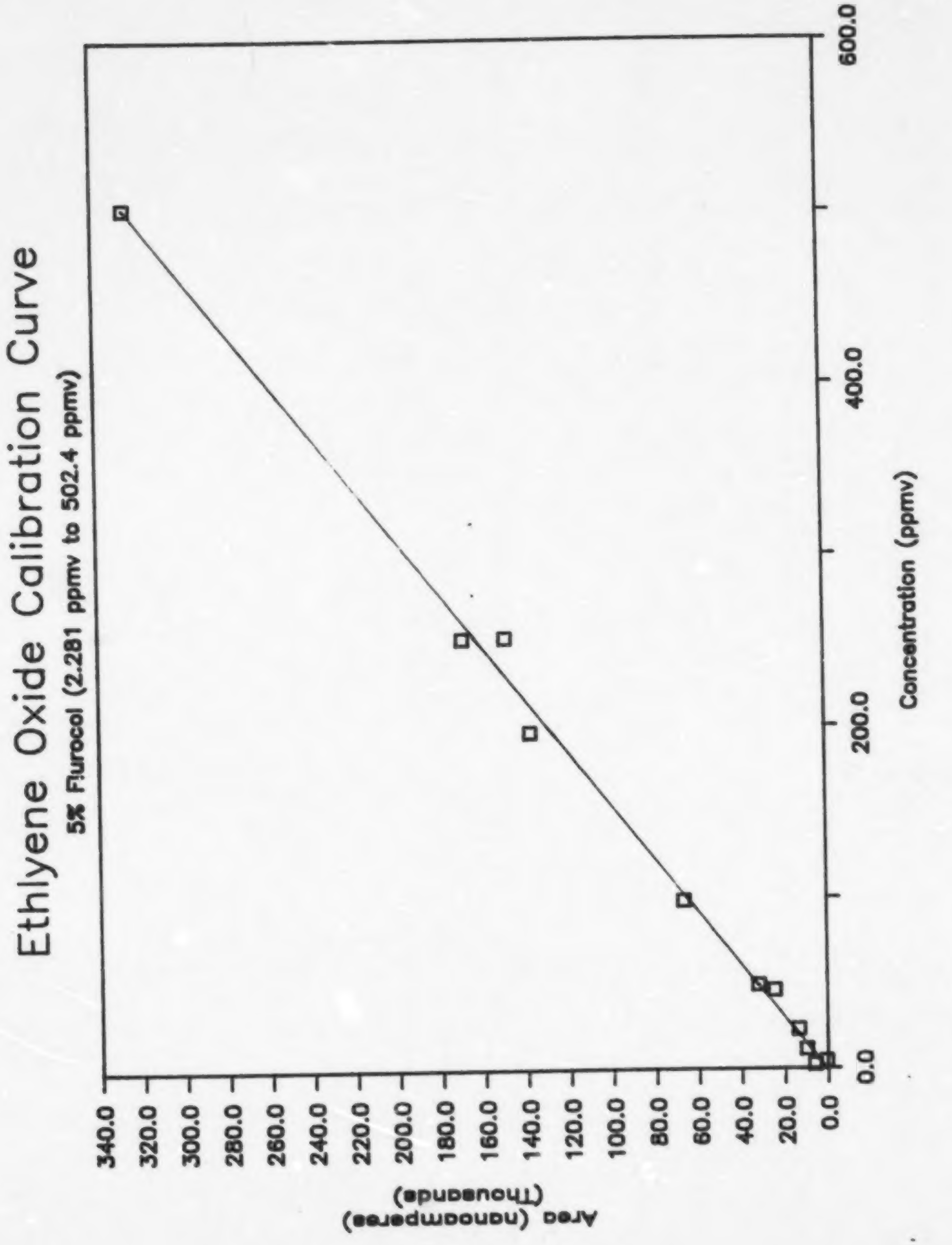


Figure B-2. Ethylene oxide calibration curve at the low part-per-million level on the 5% Fluorcol column.

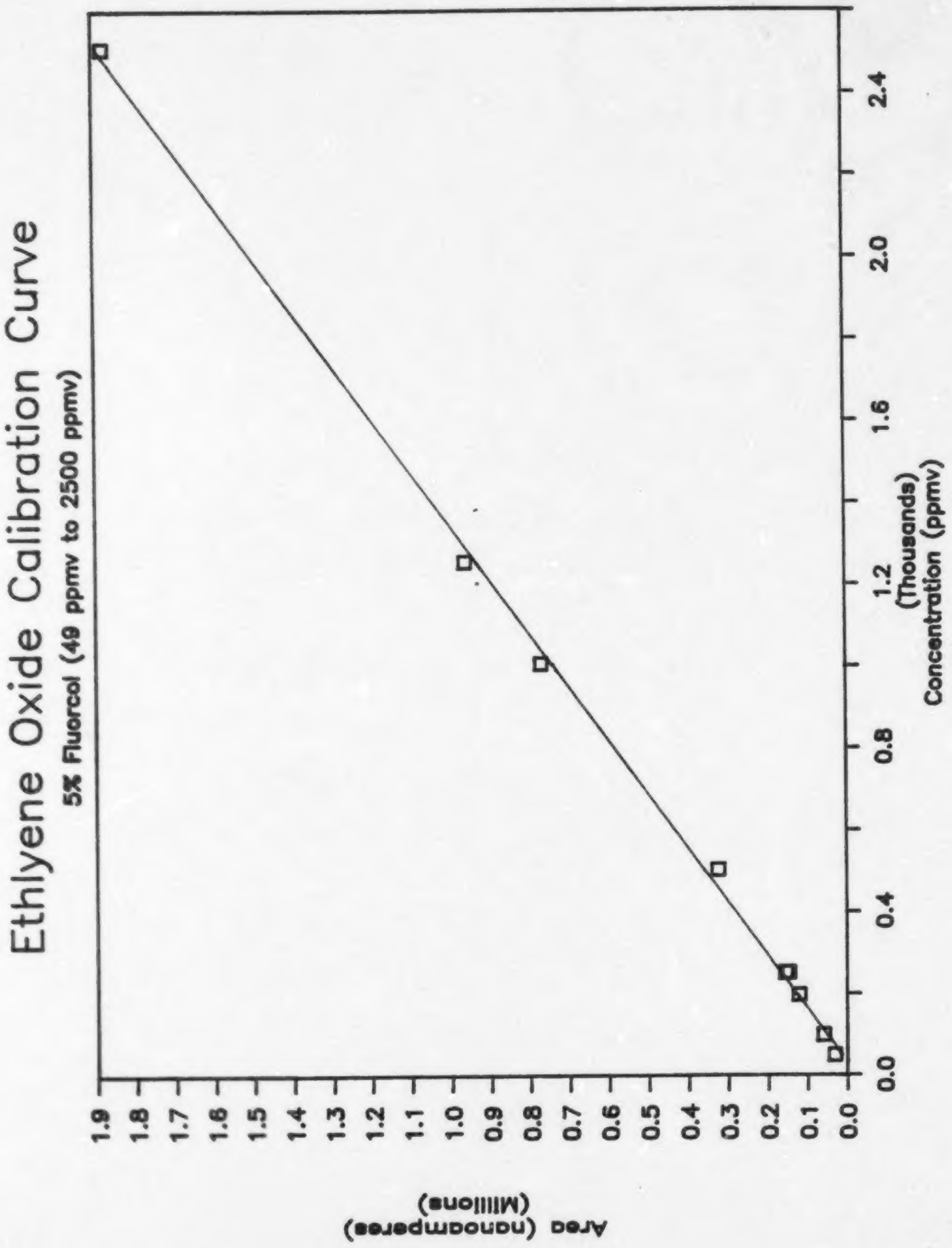


Figure B-3. Ethylene oxide calibration curve at the high part-per-million level on the 5% Fluorcol column.

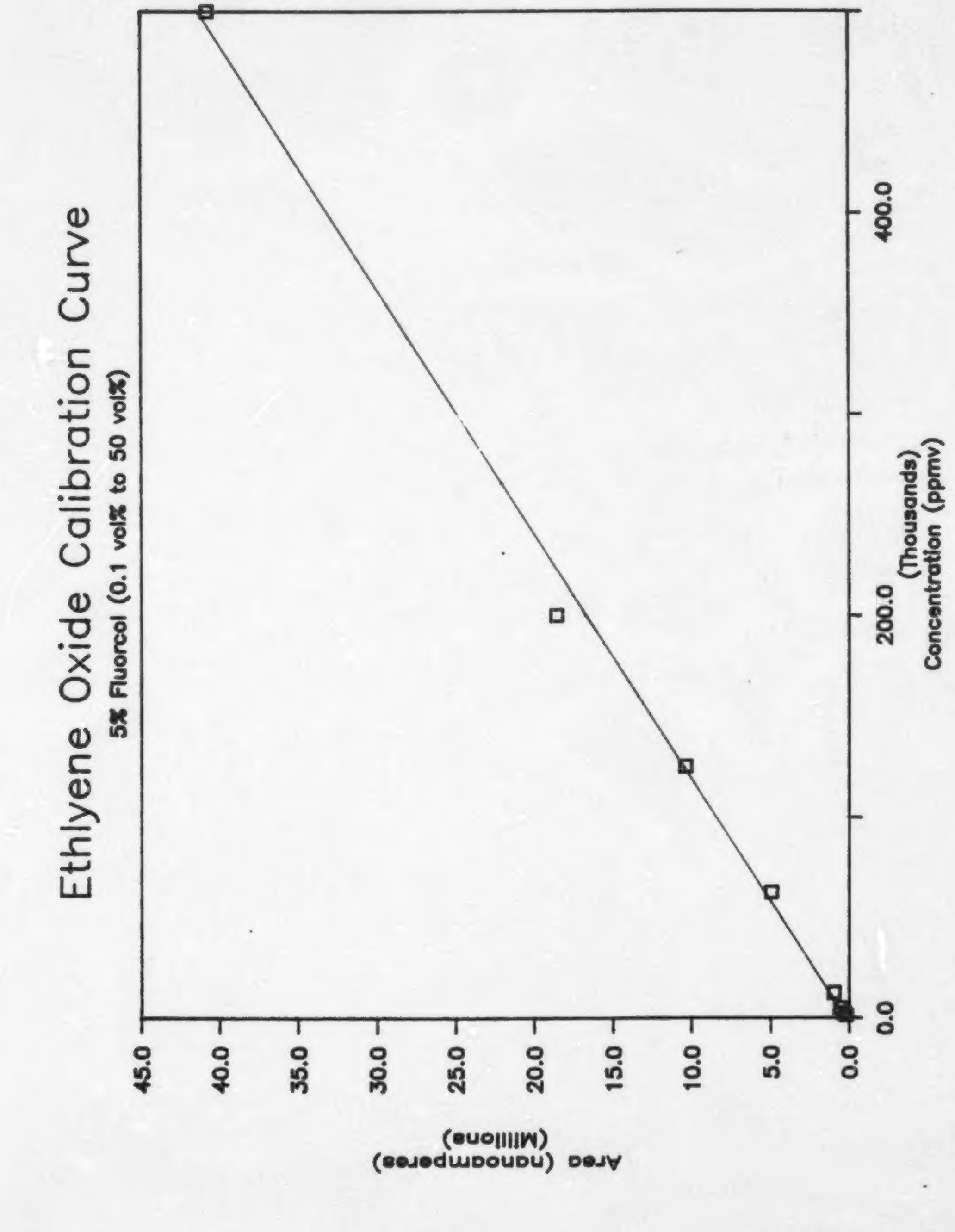


Figure B-4. Ethylene oxide calibration curve at the volume percent level on the 5% Fluorcol column.

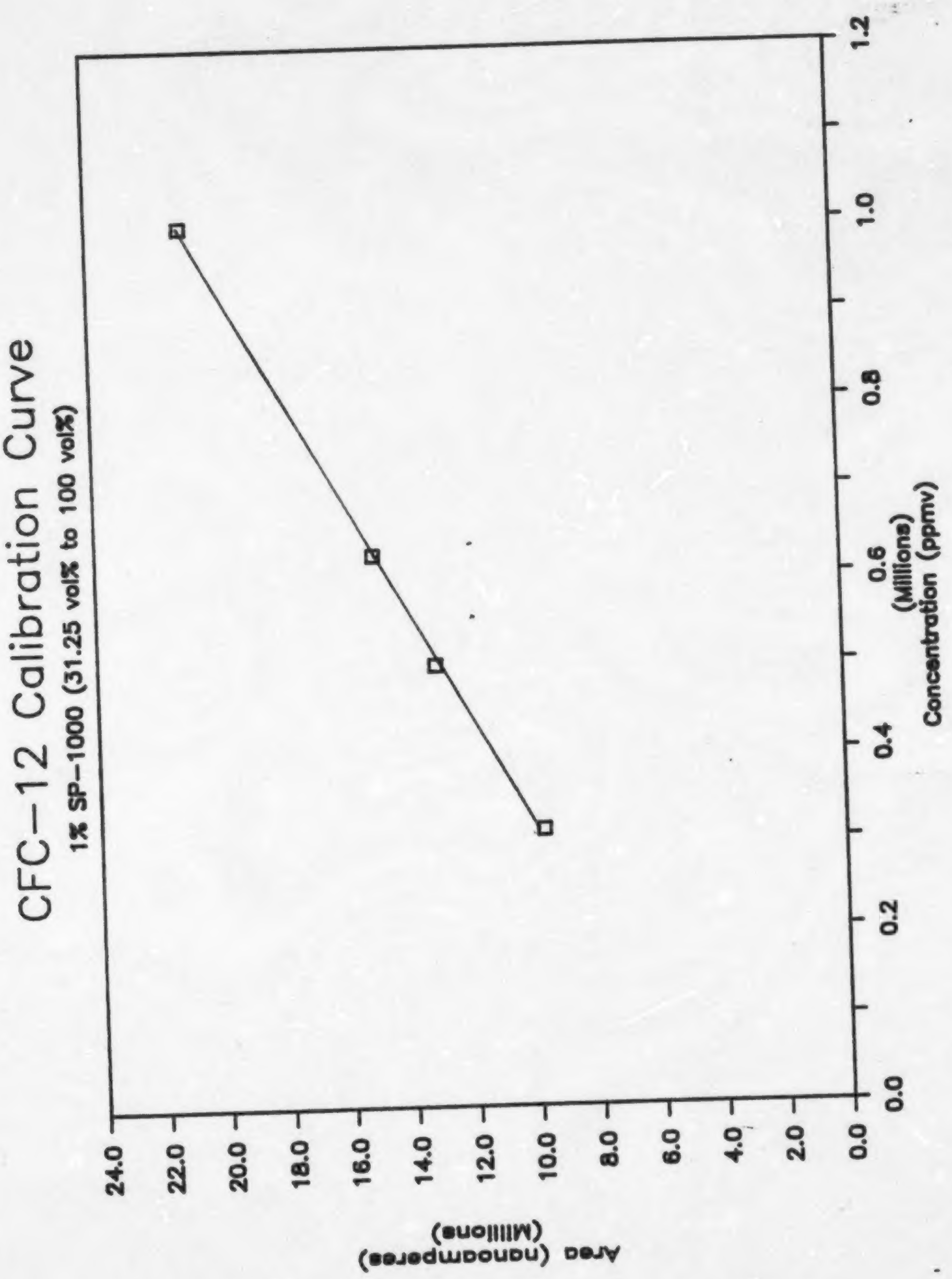


Figure B-5. Dichlorodifluoromethane calibration curve on the 1% SP-1000 column.

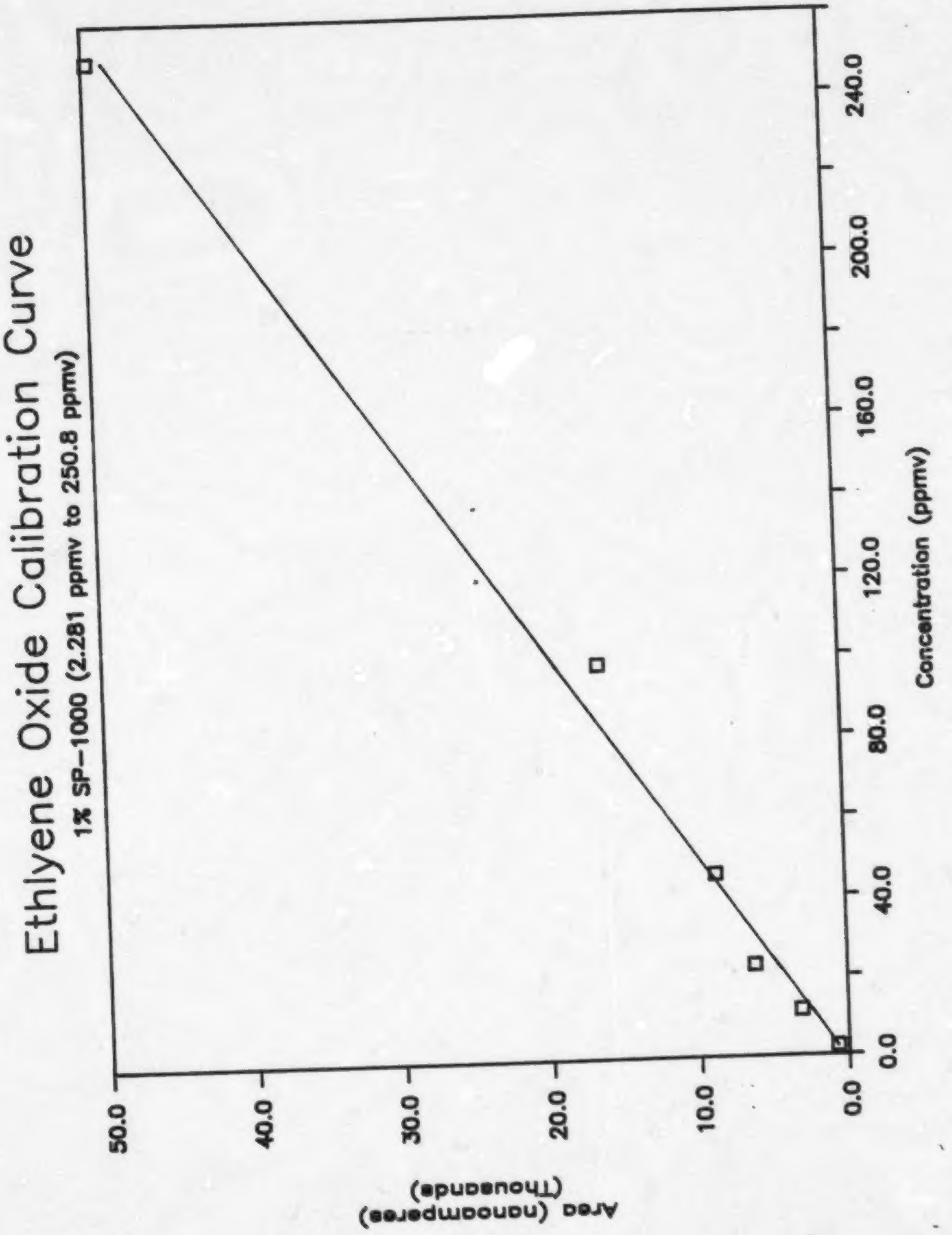


Figure B-6. Ethylene oxide calibration curve at the low part-per-million level on the 1% SP-1000 column.

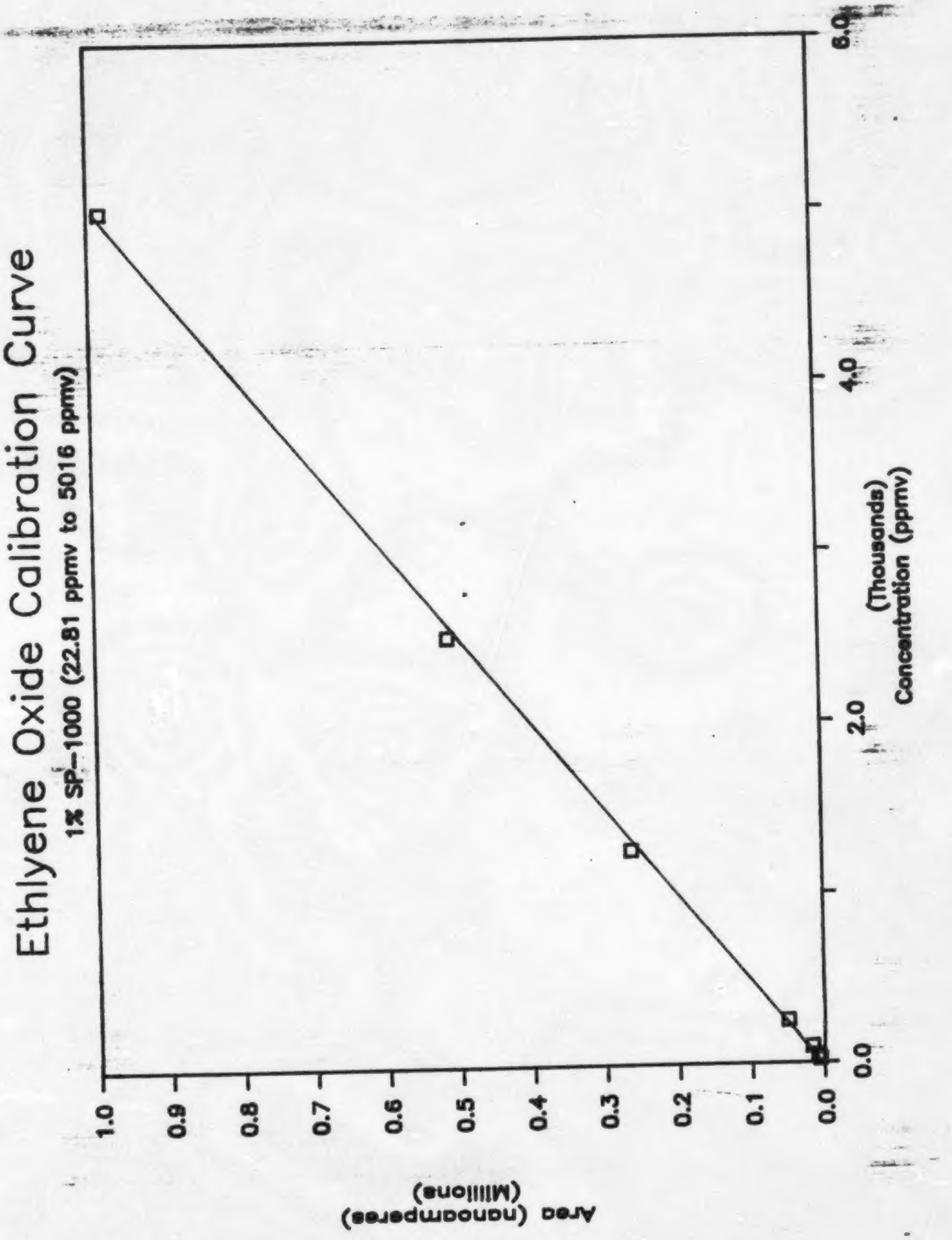


Figure B-7. Ethylene oxide calibration curve at the high part-per-million level on the 1% SP-1000 column.

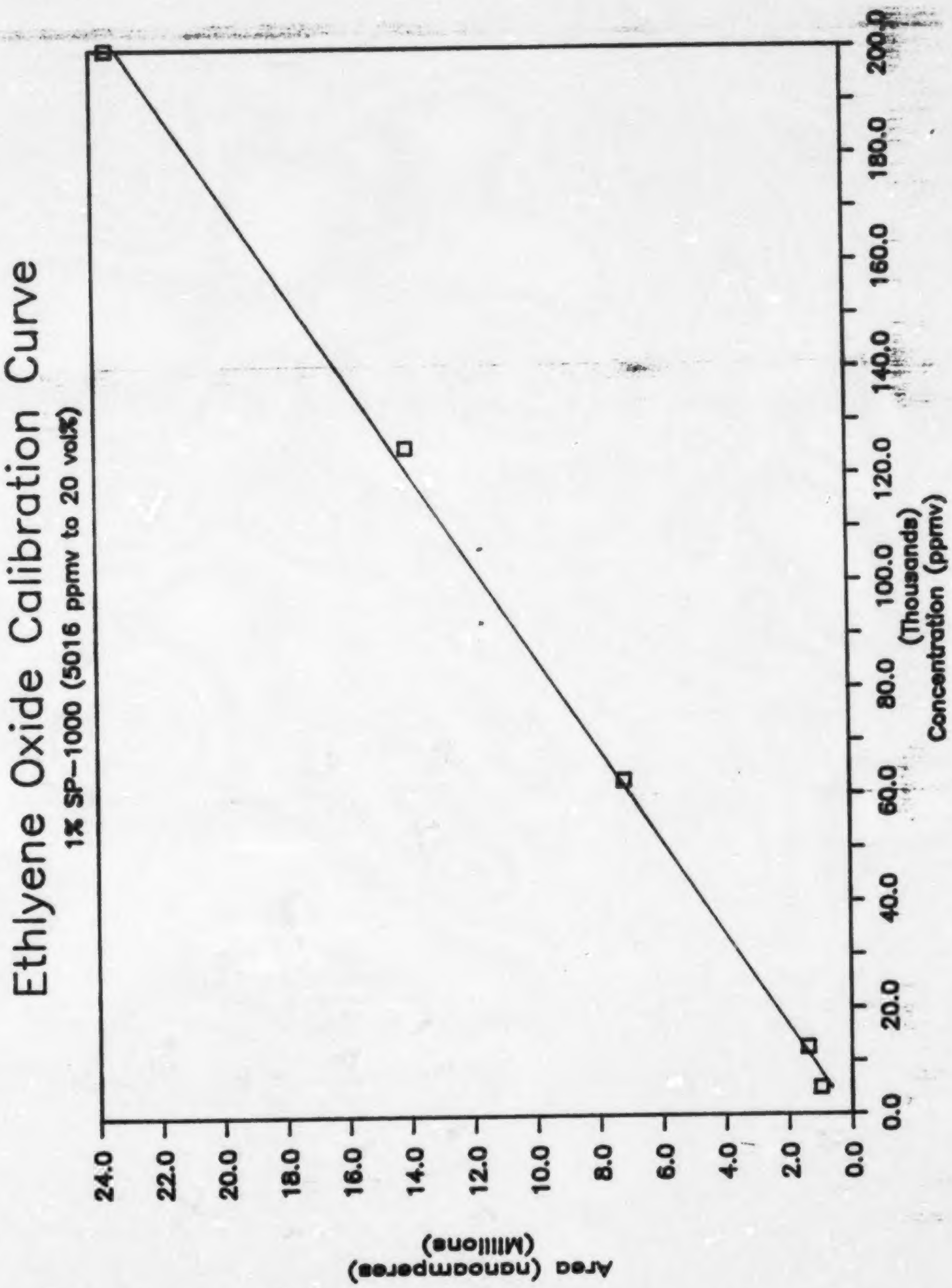


Figure B-8. Ethylene oxide calibration curve at the volume percent level on the 1% SP-1000 column.

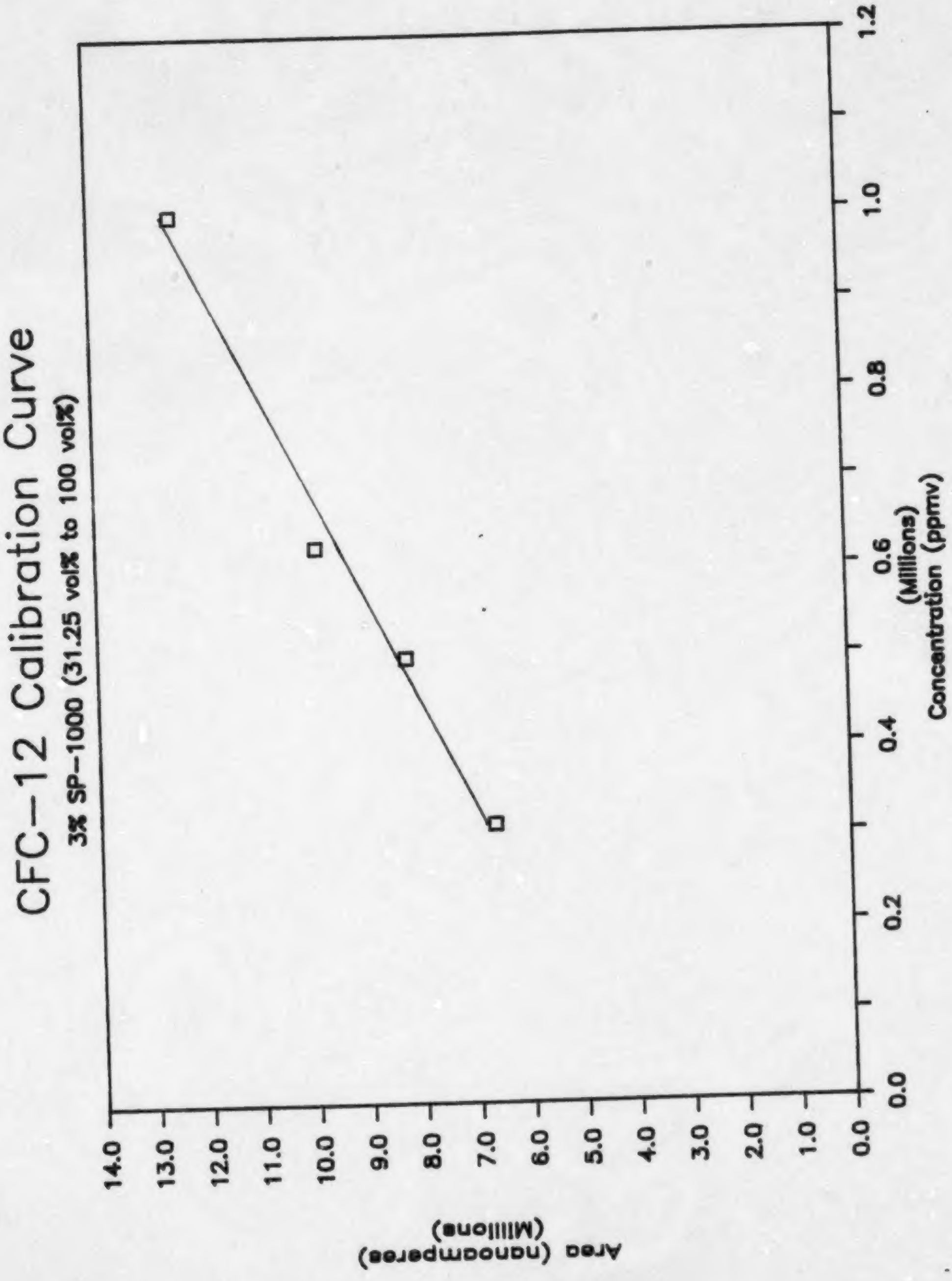


Figure B-9. Dichlorodifluoromethane calibration curve on the 3% SP-1000 column.

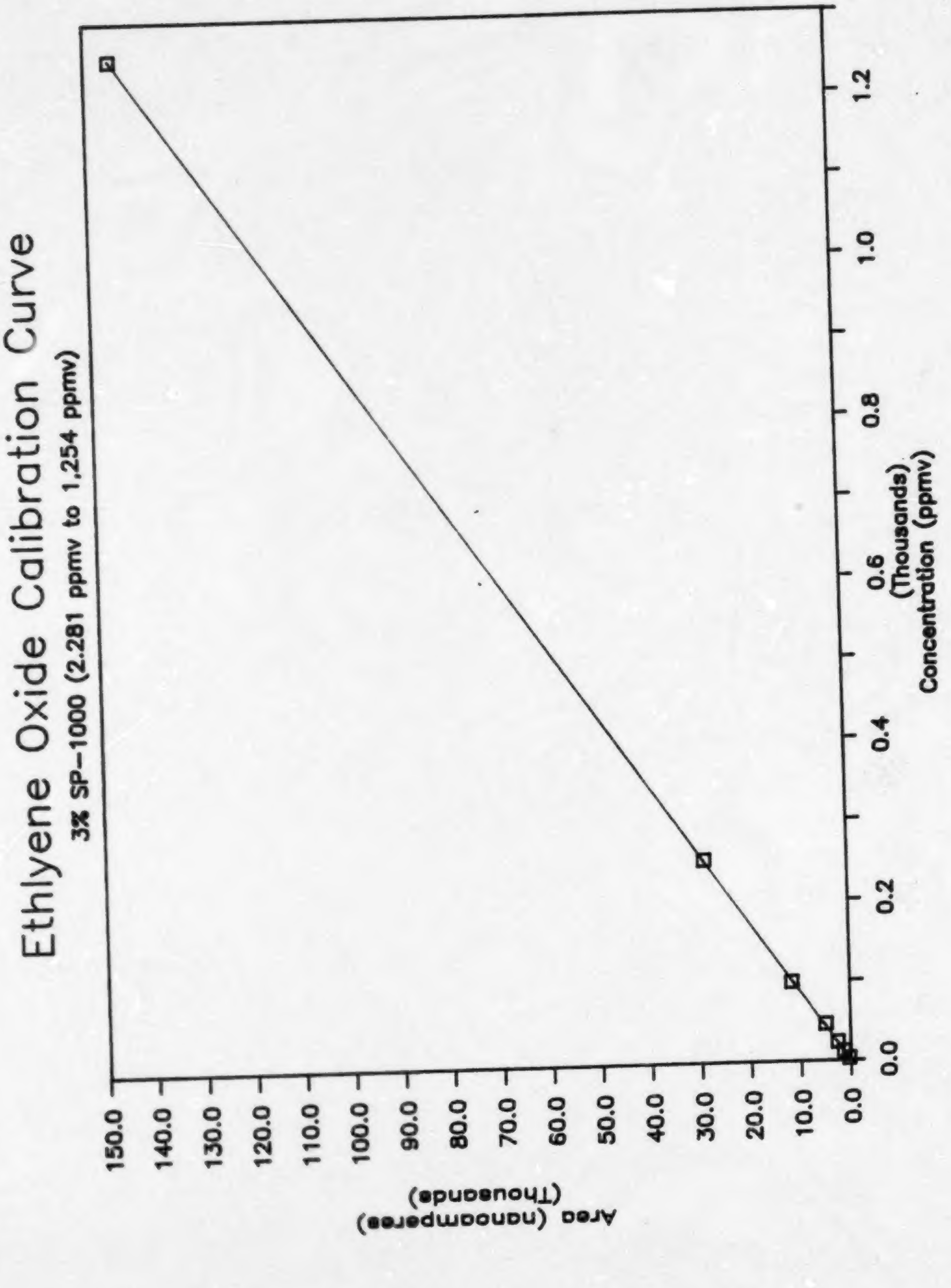


Figure B-10. Ethylene oxide calibration curve at the low part-per-million level on the 3% SP-1000 column.

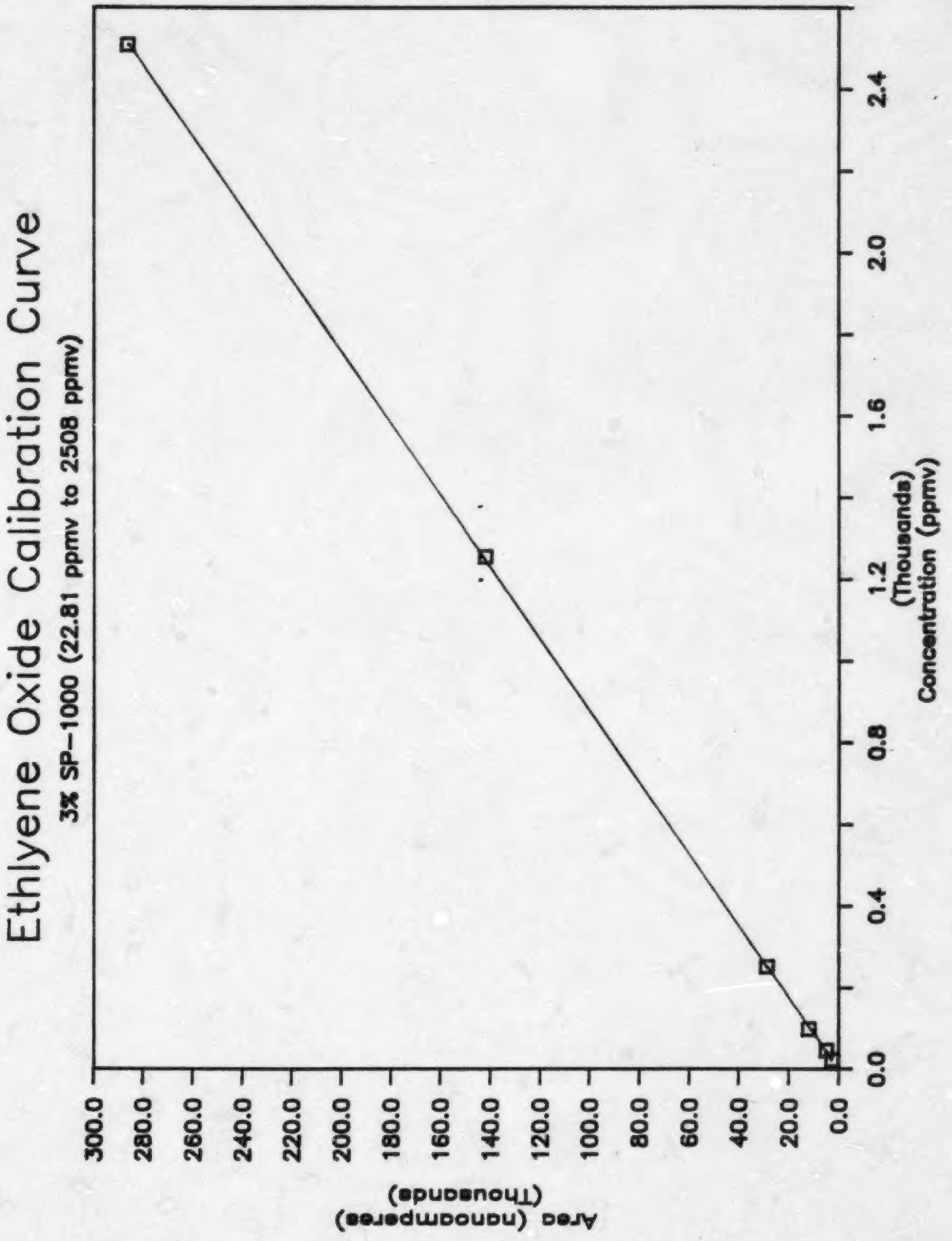


Figure B-11. Ethylene oxide calibration curve at the high part-per-million level on the 3% SP-1000 column.

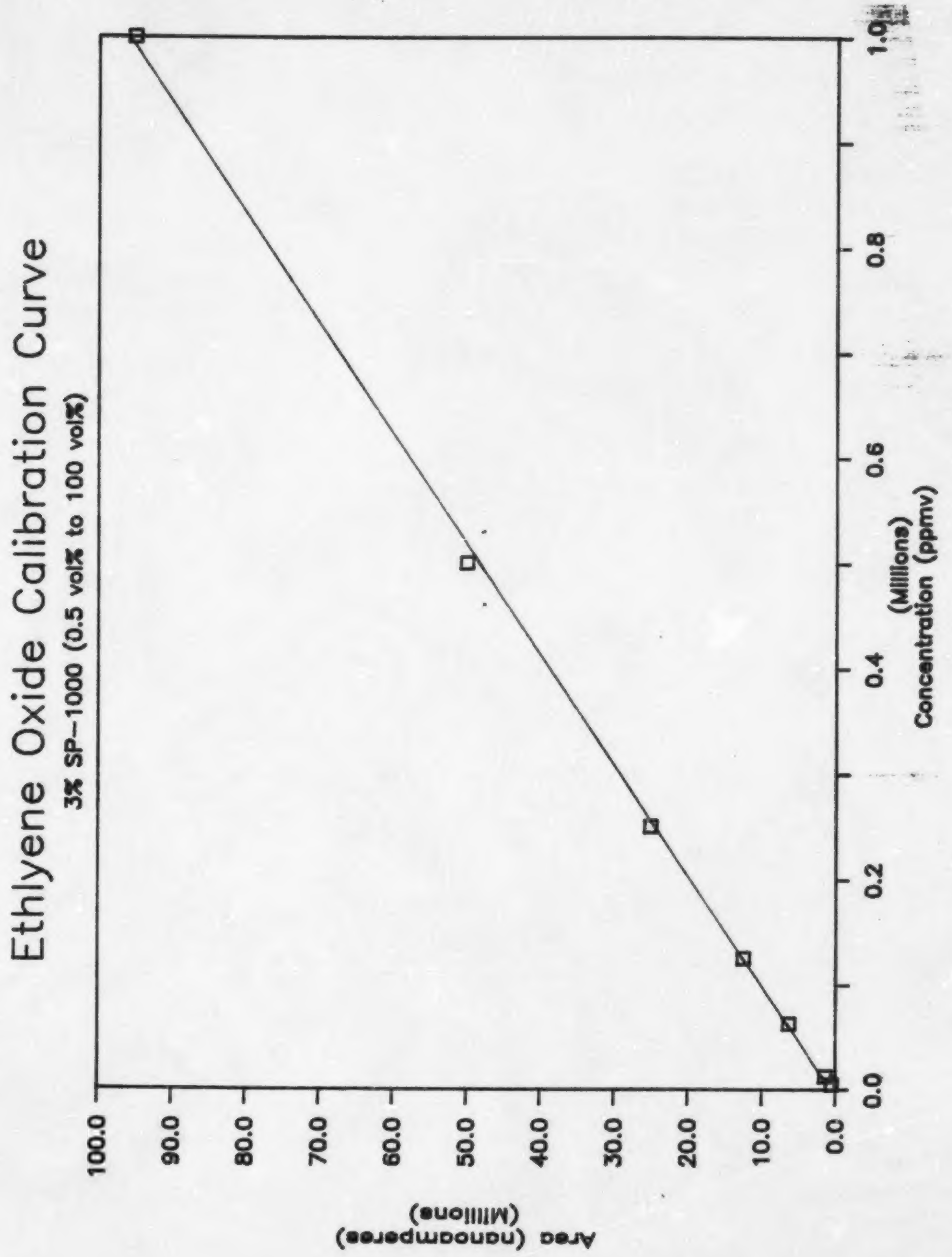


Figure B-12. Ethylene oxide calibration curve at the volume percent level on the 3% SP-1000 column.

APPENDIX C

EXAMPLE CHROMATOGRAMS

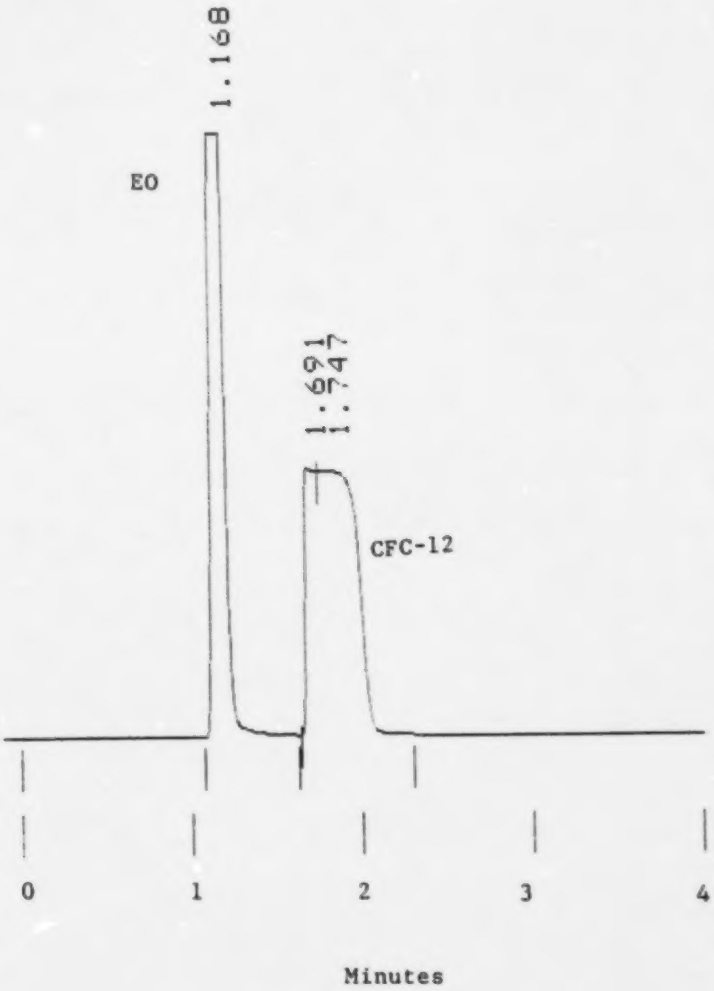


Figure C-1. Chromatogram of 62.5 vol% CFC-12 and 12.5 vol% EO on 1% SP-1000 at 65°C.

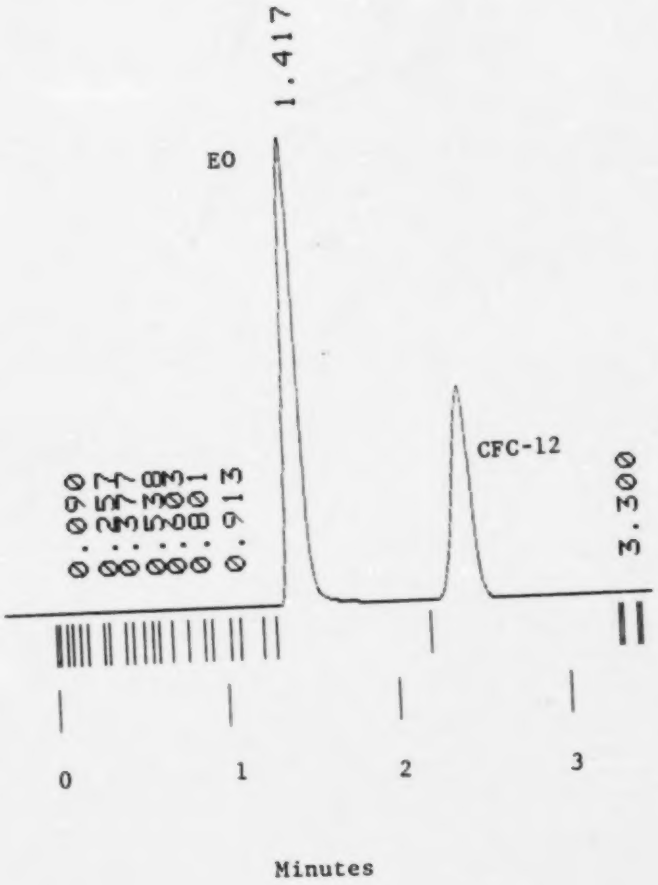
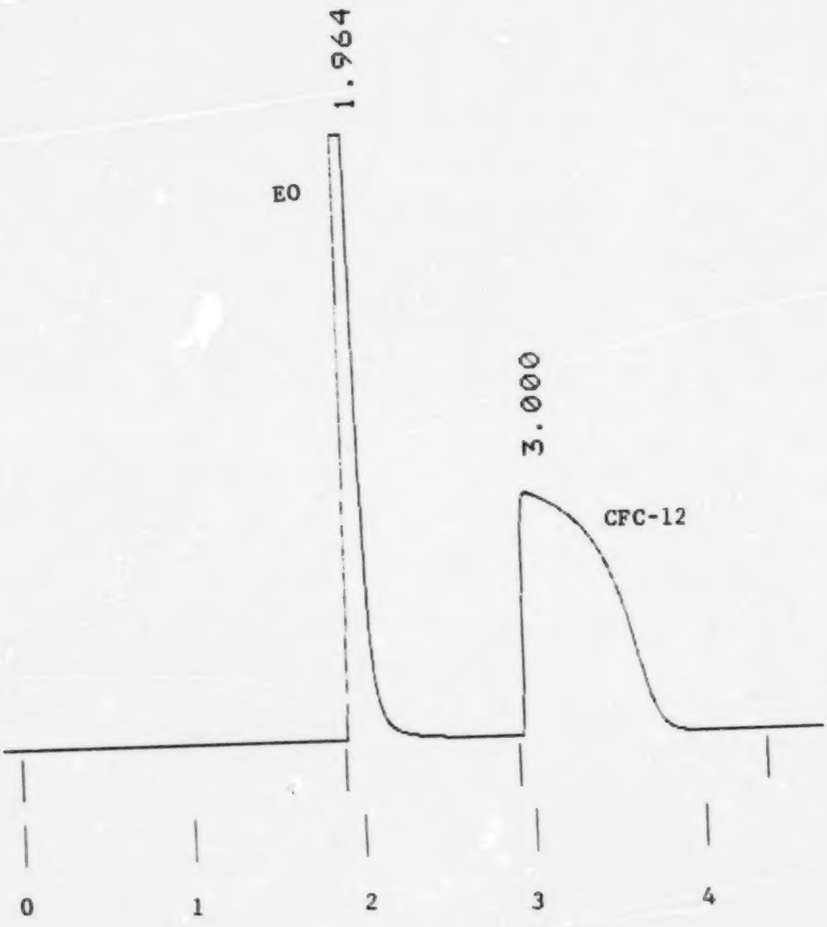


Figure C-2. Chromatogram of 6,022 ppmv CFC-12 and 2,508 ppmv EO on 1% SP-1000 at 55°C.



Minutes

Figure C-3. Chromatogram of 62.5 vol% CFC-12 and 12.5 vol% EO on 5% Fluorcol at 65°C.

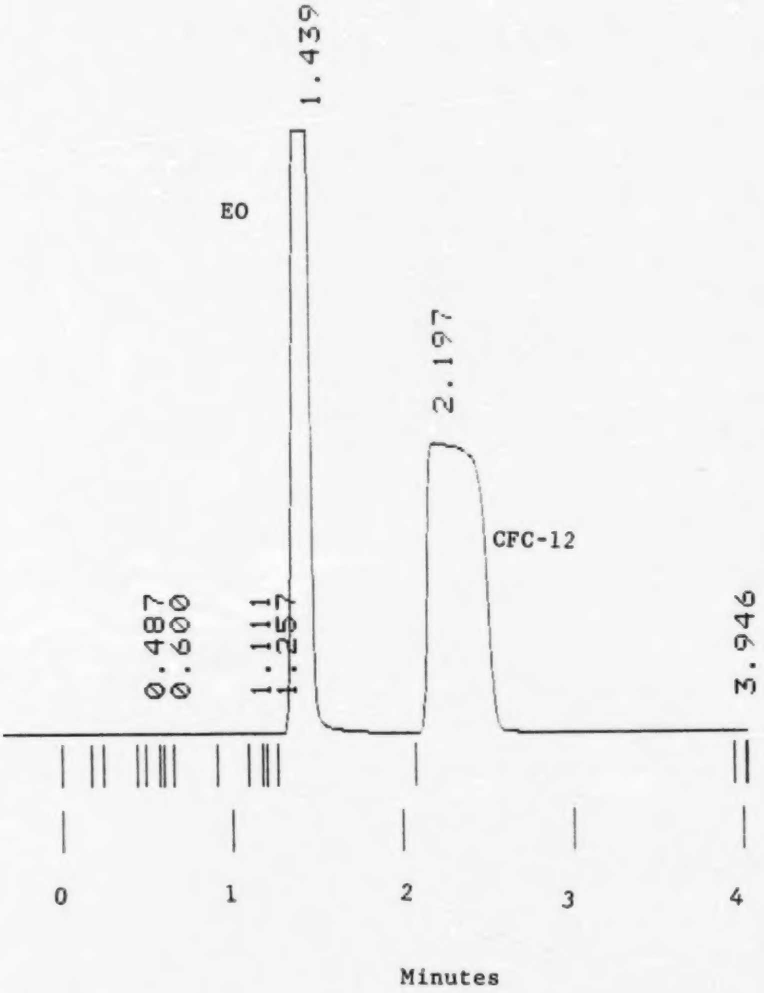


Figure C-4. Chromatogram of 62.5 vol% CFC-12 and 12.5 vol% EO on 1% SP-1000 at 55°C.

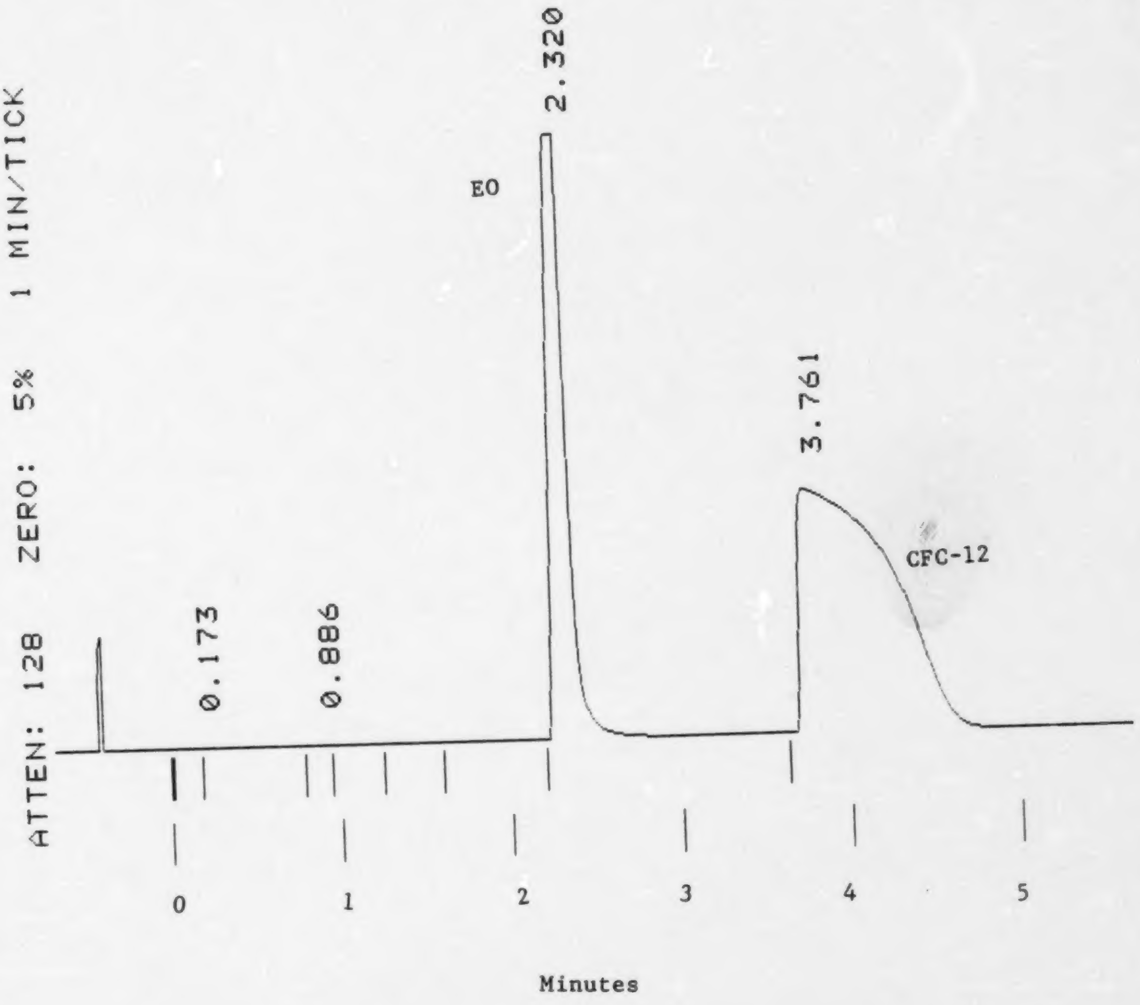


Figure C-5. Chromatogram of 62.5 vol% CFC-12 and 12.5 vol% EO on 5% Flurocol at 55°C.

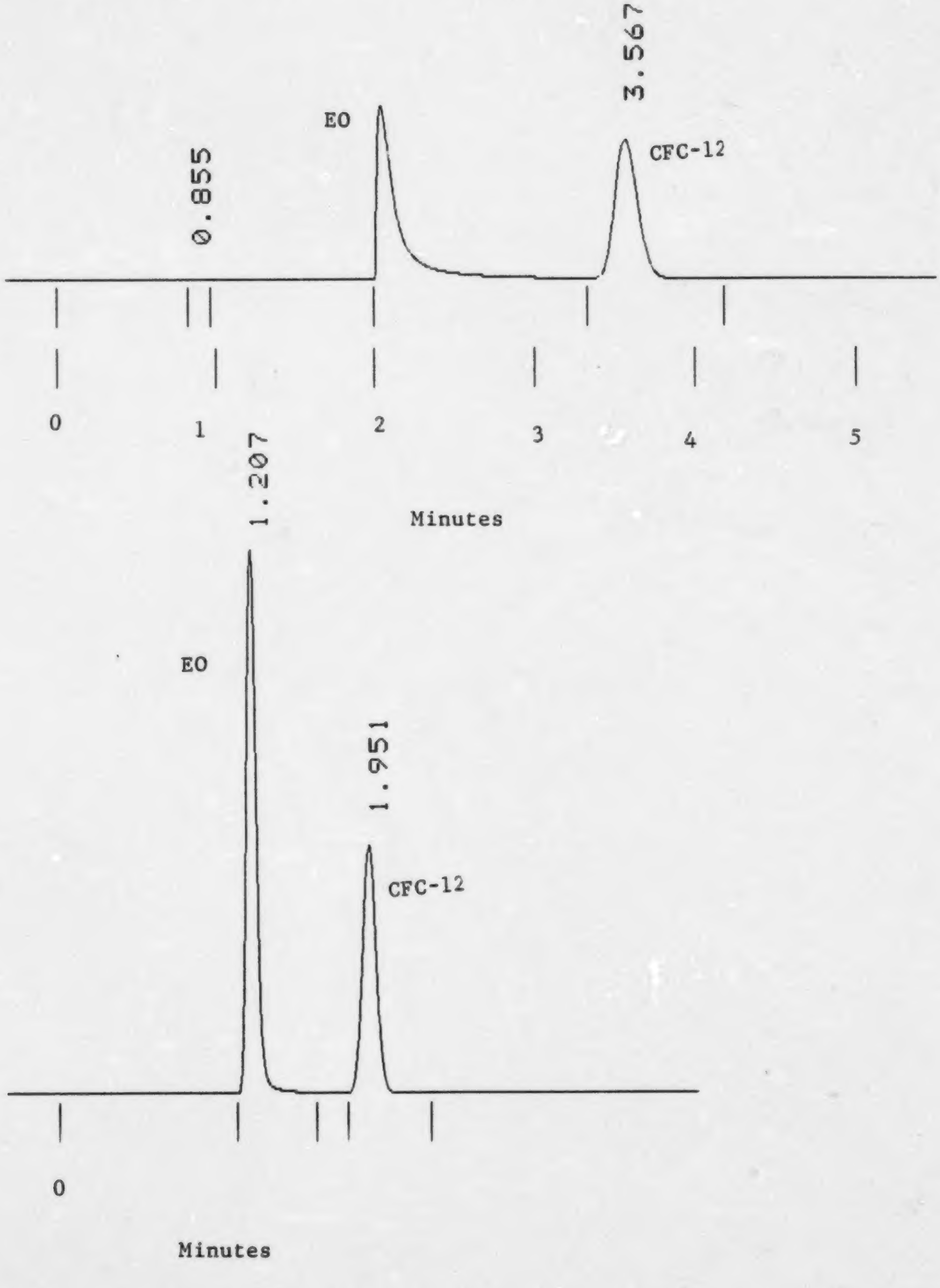


Figure C-6. Chromatograms of 6,022 ppmv CFC-12 and 2,508 ppmv EO on 5% Fluorcol (top) and 1% SP-1000 (bottom) at 65°C.

APPENDIX D

VACU-SAMPLER DESCRIPTION

MDA The Monitoring People

Vacu-Sampler

Disposable Air Sampling Collection System

- **Samples ambient air, breath, stacks, pressure lines**
- **Safe in explosive atmospheres**
- **Precise vacuum level assures good quantitative results**
- **Chemically inert**
- **Valve and septum integrity allows long shelf life before and after use**

The Vacu-Sampler is a reliable, low cost device that untrained personnel can use to collect samples of gases and vapors for laboratory analysis. This easy to use device meets the needs of both industry and government.

Vacu-Sampler is an aerosol-type can which has been completely evacuated and then back filled with nitrogen to a partial vacuum of 20 inches of mercury (246.6 cc of N_2 at standard temperature and barometric pressure). When activated, Vacu-Sampler will inhale 123.3 cc of vapors or gases (at standard temperature and pressure). The nitrogen background allows effective collection of samples in high dew point environments. During analysis of the sample, the effect of the nitrogen background is eliminated by using a dilution correction factor.*

***References**

Principe, Andrew H., "The Vacu-Sampler, A New Device for the Encapsulation of Breath and Other Gaseous Samples," presented at the 25th Annual Meeting of the Academy of Forensic Science, Las Vegas, NV, Feb. 19-23, 1973.

Principe, Andrew H., "Vacu-Sampler: A Unique Grab Sampling Device."

Miller, Robert W., et al. "Vacu-Samplers, A Simple Method for Taking Accurate Quantitative Gas and Vapor Samples Using Partially Evacuated Steel Containers," presented at the American Industrial Hygiene Conference, Minneapolis, MN, June, 1975.

The Monitoring People

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Introduction

The Vacu-Sampler is a partially evacuated rigid container, back-filled with nitrogen, with a total volume of 370 cc. When activated, it will inhale vapor or gas by equalizing the pressure difference between the inside of the container and the sampling point. When the sample is taken at standard temperature and pressure, each container will inhale 123.3 cc. The effect of sample dilution with nitrogen may be eliminated during analysis by using a correction factor, detailed below in the section entitled Quantitation.

Caution: Be sure you fully understand these instructions before using the Vacu-Sampler. If you have any questions call our toll-free number: 800-323-2000 (In Illinois 312/634-2800).

Taking A Sample

Break the tape seal and remove the safety cover to expose the white plastic actuator button. **Caution:** Do not use the Vacu-Sampler if the tape seal is broken; integrity of a sample can only be assured if the seal is intact.

Atmospheric Sampling

1. Position the intake nozzle in the space to be sampled.
2. Hold the container firmly, depress the actuator button, and hold down for 10 seconds to inhale a sample of the atmosphere.
3. Record the sample data immediately, including the ambient temperature.
4. Replace the safety cover.

Pressure Sampling

1. Place the end of the supplied Tygon tubing with partial split on the Vacu-Sampler nozzle.
2. Attach the other end of the Tygon tubing to the pressure system to be sampled. The most convenient method is to slip the Tygon tubing over a length of copper or stainless steel tubing connected to the pressure system.
3. Open the pressure system valve and allow pressurized gas to vent out of the slit in the Tygon tubing for a few seconds.
4. Hold the Vacu-Sampler firmly, depress the actuator button, and hold down for 10 seconds to inhale a sample.
5. Close the pressure system valve, remove the tubing, record the sample data, including the temperature, and replace the safety cover on the Vacu-Sampler.

Breath Sample

The most representative sample of alveolar "air" can be taken using the Tygon tubing with partial split.

1. Place the end of the Tygon tubing with partial split on the Vacu-Sampler nozzle. Place the other end of the tubing in the subject's mouth.
2. Instruct the subject to begin exhaling into the tubing. Approximately three seconds after the subject begins, and while the subject continues to exhale, hold the Vacu-Sampler firmly, depress the actuator button, and hold down for 10 seconds to inhale a sample.
3. Remove the tubing, record the sample data, and replace the safety cover. Record 34° C. (normal body temperature) as the sample temperature.

Analyzing A Sample

Gas chromatography, or another technique may be used to analyze the sample. Be sure the safety seal is in place, and that the Vacu-Sampler container is carefully packed for shipment.

Extracting A Sample

1. Remove the safety cover. Carefully remove the white plastic actuator assembly.
2. With a sharp, pointed punch, such as an ice pick or small finishing nail, carefully puncture the metal can-top seal. Make a hole just large enough to accept the needle of a gas-tight syringe. Carefully remove the punch; if the rubber septum below the metal seal is invaded by the punch, the septum will reseal when the punch is removed, maintaining the integrity of the sample.
3. Insert the syringe needle through the septum and withdraw a sample for laboratory analysis.

Quantitation

Accurate analysis requires eliminating the dilution of the sample from nitrogen. The Dilution Correction Factor is a function of sample temperature when taken related to sample temperature when analyzed in accordance with gas laws:

$$\text{Dilution Correction Factor} = 3\,\frac{T_1}{T_2}$$

where T_1 = Sample Temperature (°K) at time of sampling
T_2 = Vacu-Sampler Temperature (°K) at time of analysis

Example

Assume that we sample acetone vapor from a room with an ambient temperature of 35° C. Following the analysis procedures for gas chromatography, utilizing standards, we find that the sample from the Vacu-Sampler gives us an apparent value of 25 ppm when analyzed at 21° C.

Using the formula above:

$$\text{since } T_1 = 273 + 35 = 308°\text{K}.$$
$$\text{and } T_2 = 273 + 21 = 294°\text{K}.$$
$$\text{Dilution Factor} = 3 \times \frac{308}{294} = 3.14$$

Correcting the apparent value:

$$25 \text{ ppm} \times 3.14 = 78.5 \text{ ppm}$$

More detailed quantitative instructions are included with each case of Vacu-Samplers.

Cat. No. 970214 12/85 10M

APPENDIX E

GRAB SAMPLING DATA SHEETS

1 of 6

ETHYLENE OXIDE SAMPLING DATA SHEET

DATE 6/21

RUN ID 1

SAMPLER WPG/PRL/JLS

SCHEDULE:

INITIAL EXHAUST 1 bag/min

AIR WASHES 3/wash

FACILITY Burron

BAROMETRIC PRESSURE 30.47

		13:03			TEMPERATURE (F)				PERCENT	Ps	
TIME	EXHAUST #	START	CONTAINER	ID	VENT	t'	PUMP	HEAT TRACE	OXYGEN	(IN H₂O)	NOTES
13:04	I	13:04	BAG	1	90	79	163	103	19.5	.2	
13:05	↓	↓	↓	2	92	77	163	162	20.0	.2	
:06				3	93	76	162	102	14.0	.2	
:07				4	94	75	160	101	4.0	.4	
:08				5	95	74	159	100	0.5	.5	
:09				6	96	74	158	100	0.0	.4	
:10				7	98	73	157	100	0.0 ~~0.4~~	0.4	
:11				8	98	73	156	100	0.0	.2	
:12				9	100	72	155	101	0.0	.2	
:13				10	100	72	155	102	0.0	.1	
:14				11	100	72	154	102	0.0	.1	

2 of 6

ETHYLENE OXIDE SAMPLING DATA SHEET

DATE 6/21

RUN ID 1

SAMPLER

SCHEDULE:

INITIAL EXHAUST 1

AIR WASHES 65

FACILITY Becton

BAROMETRIC PRESSURE

TIME	EXHAUST #	START	CONTAINER	ID	TEMPERATURE (F) VENT	t'	PUMP	HEAT TRACE	PERCENT OXYGEN	Ps (IN Hg)	NOTES
13:15	I	13:03	BAG	12	100	72	159	102	0.0	.1	
:16	↓	↓	↓	13	100	71	153	103	0.0	.1	
:17				14	100	72	153	103	0.0	.1	
:18				15	100	72	153	103	0.0	.1	
:19				16	100	72	152	103	0.0	.05	
:20				17	99	72	151	103	0.0	.05	
:21				18	98	73	150	103	0.0	.05	
:22				19	97	73	150	102	0.0	.05	
:23				20	97	73	150	102	0.0	—	
:24				21	101	72	150	101	0.0	.4	← Prevac for another unit
:25				22	104	71	150	102	0.0	.4	

3 of 6

ETHYLENE OXIDE SAMPLING DATA SHEET

DATE 6/21/88

RUN ID 1

SAMPLER

SCHEDULE:

INITIAL EXHAUST 1

AIR WASHES 5

FACILITY Burron

BAROMETRIC PRESSURE

					TEMPERATURE (F)				PERCENT	Ps	
TIME	EXHAUST #	13:03 START	CONTAINER	ID	VENT	t'	PUMP	HEAT TRACE	OXYGEN	(IN Hg)	NOTES
13:26	I	~~23~~	BAG	23	106	70	149	101	0.0	.3	
13:27	↓		↓	24	107	69	148	100	0.0	.3	
			↓		END	of	Initial	Exhaust			
	AW-1	13:58	BAG								
14:00			↓	—					14.0		Looks Like residual O_2
14:00:30			↓	—					15.5		from earlier prevac.
14:03			↓	—					15.0		— Air Inbleed for #3 sterilizer
14:08	AW-1	14:08	BAG	—	121				0.0		Airwash #1 Starts
14:10			↓	—	121	71	151	103	0.0		
14:11			↓	—					1.0		

Not AW1

4 of 6

ETHYLENE OXIDE SAMPLING DATA SHEET

DATE 6/21
RUN ID 1
SAMPLER

SCHEDULE:
INITIAL EXHAUST 1
AIR WASHES 65

FACILITY Burron
BAROMETRIC PRESSURE

					TEMPERATURE (F)				PERCENT	Ps	
TIME	EXHAUST #	START	CONTAINER	ID	VENT	t'	PUMP	HEAT TRACE	OXYGEN	(IN Hg)	NOTES
14:12	AW-1	14:08	BAG	—	122	69	151	103	6.0	.4	
14:14	↓	↓	↓	#25	122	68	152	109	8.0	.3	
14:17				#26	122	68	154	115	14.0	.1	
14:20				#27	120	69	155	119	18.0	.05	
14:24	[illegible]	END	AW-1	(noticeable)			→				14:30 - Bill called said [illegible] end of wash - low flow, not noticeable w/o vane anemometer
14:39	AW-2	4 wk 2 14:39	BAG	—	112	73	159	12'	16.0	.2	- Fyrite fluid change.
14:41		↓	↓	—					15.0		
14:44				#28	121	69	161	124	16.0	.2	
14:47				#29	119	68	161	123	16.5	.1	
14:50				#30	118	69	161	122	16.5	.1	

15:00 approx. end of wash per control panel

5 of 6

ETHYLENE OXIDE SAMPLING DATA SHEET

DATE 6/21/88
RUN ID 1
SAMPLER
SCHEDULE:
INITIAL EXHAUST 1
AIR WASHES 6
FACILITY Burron
BAROMETRIC PRESSURE

TIME	EXHAUST #	START	CONTAINER	ID	TEMPERATURE (F) VENT	t'	PUMP	HEAT TRACE	PERCENT OXYGEN	Ps (IN Hg)	NOTES
15:09	AW-3	15:09	Bag						15.5		
15:14	↓	↓	↓	31	110	70	163	120	16.0	.1	
15:17				32	109	77	164	121	16.0	.1	
15:20				33	108	86	165	123	16.5 ~~to Out~~	—	Ambient fyrite √ = O_2 @ 17.0 Changed sol'n again
15:38:30	AW-4	15:38	Bag								
15:44	↓	↓	↓	34	110	91	168	128	19.5	.1	
15:47				35	110	89	168	118	20	.1	
15:50 wRL ~~16:00~~				36	107	90	168	111	20	.1	

6 of 6

ETHYLENE OXIDE SAMPLING DATA SHEET

DATE 6/21

RUN ID 1

SAMPLER WPK/PTL/JES

SCHEDULE:

INITIAL EXHAUST 1

AIR WASHES 5

FACILITY Burton

BAROMETRIC PRESSURE ______

16:08

					TEMPERATURE (F)						
TIME	EXHAUST #	START	CONTAINER	ID	VENT	t'	PUMP	HEAT TRACE	PERCENT OXYGEN	Ps (IN Hg)	NOTES
16:13	AW-5	↓	BAG	37	106	92	170	111	20	.2	
16:16	↓		↓	38	108	90	170	111	↓	.1	
16:19				39	109	90	171	113		.1	

1 of 5

ETHYLENE OXIDE SAMPLING DATA SHEET

DATE	6/22	SCHEDULE:		FACILITY	Burron Med
RUN ID	2	INITIAL EXHAUST	1	BAROMETRIC PRESSURE	30.48
SAMPLER	WPG/PTL	AIR WASHES	5		

TIME	EXHAUST #	START	CONTAINER	ID	TEMPERATURE (F) VENT	t'	PUMP	HEAT TRACE	PERCENT OXYGEN	Ps (IN Hg)	NOTES
	Init.	10:00									
10:05	↓	↓	BAG	40	97	63	141	106	15.0	.2	
10:11	↓	↓	BAG	41	92	61	139	106	1.0	.1	
10:12			V	200A 200B							
10:13			Sy	101 102							
10:17			BAG	42	89	62	138	106	0.5	—	
10:18			Sy	103 104							
Evac End @			10:56								Evacuated #2 ster. between
	AW-1	10:03									In & AW-1 (Pre sterilize evac)
10:08	↓	↓	BAG	43	102	62	139	114	14 ~~[illegible]~~	.2	
10:12	↓	↓	BAG	44	101	61	141	115	19 ↓	.1	

Sy = Syringe

V = Vaccsampler Can

2 of 5

ETHYLENE OXIDE SAMPLING DATA SHEET

DATE 6/22

RUN ID 2

SAMPLER WPC/PTL

SCHEDULE:

INITIAL EXHAUST 1

AIR WASHES 5

FACILITY Burton

BAROMETRIC PRESSURE 30.48

					TEMPERATURE (F)						
TIME	EXHAUST #	START	CONTAINER	ID	VENT	t'	PUMP	HEAT TRACE	PERCENT OXYGEN	Ps (IN Hg)	NOTES
11:13	AW-1	11:03	Sy	105 106							
11:14	↓	↓	V	201A 201B							
11:15			BAG	45	98	61	140	113	19.5	.1	
11:16			Sy	107 108							
11:26	END AIR WASH #1										
	AW-2	11:36									
11:41	↓	↓	Bag	46	100	62	140	116	20	.1	
11:45			Bag	47	99	61	140	115	↓	—	
11:46			Sy	109 110							
11:47			V	202 A 202 B							

3 of 5

ETHYLENE OXIDE SAMPLING DATA SHEET

DATE 6/22
RUN ID 2
SAMPLER WPG/PTK

SCHEDULE:
INITIAL EXHAUST 1
AIR WASHES 5

FACILITY Burron
BAROMETRIC PRESSURE 30.48

					TEMPERATURE (F)						
TIME	EXHAUST #	START	CONTAINER	ID	VENT	t'	PUMP	HEAT TRACE	PERCENT OXYGEN	Ps (IN Hg)	NOTES
11:49	ΔW-2	11:36	Bag	48	96	62	141	115	20	—	
11:50	↓	↓	Sy	111 112					↓		
					END	AW-2	@ 11:55				(Interim Air Evac from steriTizer #2)
	AW-3	12:06 ~~12:04~~									
12:~~09~~ 11	↓	↓	BAG	49	103	62	142	116	20	.2	
12:~~13~~ 15	↓	↓	Bag	50	102	61	144	115	↓	.1	
12:~~14~~ 16			Syr	113 114							
12:~~15~~ 17			V	203 A 203 B							
12:~~17~~ 19			Bag	51	102	62	144	117		—	
12:~~18~~ 20			Syr	115 116							

End AW-3 @ 12:26

4 of 5

ETHYLENE OXIDE SAMPLING DATA SHEET

DATE 6/22
RUN ID 2
SAMPLER DTL/WPG

SCHEDULE:
INITIAL EXHAUST 1
AIR WASHES 5

FACILITY Burton
BAROMETRIC PRESSURE 30.48

TIME	EXHAUST #	START	CONTAINER	ID	TEMPERATURE (F) VENT	t'	PUMP	HEAT TRACE	PERCENT OXYGEN	Ps (IN Hg)	NOTES
	AW-4	12:37									
12:42	↓	↓	BAG	52	101	63	146	113	20.5	.1	
12:46			BAG	53	101	63	145	113	↓	.1	
:47			Sy	117 118							
:48			✓	204A 204B							
12:50			Bag	54	100	64	146	114		—	
12:51			Sy	119 120							
				End@		:55					
	AW-5	13:06									
13:11			BAG	55	106	77	147	115	20.6		
13:15			BAG	56	104	76	145	113	↓		

5 of 5

ETHYLENE OXIDE SAMPLING DATA SHEET

DATE 6/22
RUN ID 2
SAMPLER PTL/WPG
SCHEDULE:
INITIAL EXHAUST 1
AIR WASHES 5
FACILITY Burron
BAROMETRIC PRESSURE 30.48

TIME	EXHAUST #	START	CONTAINER	ID	TEMPERATURE (F) VENT	t'	PUMP	HEAT TRACE	PERCENT OXYGEN	Ps (IN Hg)	NOTES
13:16	AW-5	13:06	Sy	121 122					20.0	.1	
13:17	↓	↓	V	205A 205B					↓		
13:19	↓	↓	Bag	57	104	79	148	116		.1	
13:20	↓	↓	Sy	123 124							
				13:26 –		END					

206
58

1 of 4

ETHYLENE OXIDE SAMPLING DATA SHEET

DATE 6/23/88
RUN ID 3
SAMPLER PTL/WPG

SCHEDULE:
INITIAL EXHAUST 1 WPG
AIR WASHES ~~8~~ 4

FACILITY Burron
BAROMETRIC PRESSURE

100% ETO Run

TIME	EXHAUST #	START	CONTAINER	ID	TEMPERATURE (F) VENT	t'	PUMP	HEAT TRACE	PERCENT OXYGEN	Ps (IN Hg)	NOTES
	Initial	11:45									
11:50	↓		Bag	58	77	68	150	120	20	.3	
11:56			Bag	59	80	66	151	124	19.5	.2	
11:57			V	206-A 206-B							
11:58			Sy	101A 103A							
12:02			Bag	60	79	66	153	125	20.0	—	
12:03			V	207-A 207-B							
						END	@	12:10			
	AW-1	12:58							19.5		
13:03	↓	↓	BAG	61	86	65	156	129	20.5	.2	
13:07	↓	↓	BAG	62	89	65	157	126	20.5	.1	

2 nitrogen purges prior to Loading EtO. O_2 #s don't make sense. Also ~~Nitrogen inbleeds as~~ opposed to Air inbleeds w/ 12/88.
↓
WRONG
↓
Air inbleeds

V = Vacu-sampler can
Sy = Syringe

2 of 4

ETHYLENE OXIDE SAMPLING DATA SHEET

DATE 6/23/88

RUN ID 3

SAMPLER PTL/WPG

SCHEDULE:

INITIAL EXHAUST 1

AIR WASHES 5

FACILITY Burron

BAROMETRIC PRESSURE ______

TIME	EXHAUST #	START	CONTAINER	ID	TEMPERATURE (F) VENT	t'	PUMP	HEAT TRACE	PERCENT OXYGEN	Ps (IN Hg)	NOTES
13:08	AW-1		V	208 A 208 B	89	65	158	127		.1	
13:09	↓	↓	Sy	109 A 111 A							
13:11			BAG	63	89	65	158	127	20.5	.1	
13:12			V	209 A 209 B							
		end @		13:15							
	AW-2	13:29									
13:34	↓	↓	BAG	64	88	65	157	124	20.0	.3	
13:38			Bag	65	87	65	158	122	20.5	.2	
13:39			V	210 A 210 B	87	65	156	122	↓	–	
13:40			Sy	117 A 119 A							
13:42 13:43			Bag V	66 211 A 211 B	87	65	156	122		–	

END @ 13:43

3 of 4

ETHYLENE OXIDE SAMPLING DATA SHEET

DATE 6/23/88
RUN ID 3
SAMPLER QTL/WPG

SCHEDULE:
INITIAL EXHAUST 1
AIR WASHES 5

FACILITY Burron
BAROMETRIC PRESSURE

TIME	EXHAUST #	START	CONTAINER	ID	TEMPERATURE (F) VENT	t'	PUMP	HEAT TRACE	PERCENT OXYGEN	Ps (IN Hg)	NOTES
	AW-3	13:55									
14:00	↓	↓	Bag	67	91	65	153	119	20.5	.3	
14:04			Bag	68	89	65	149	116	↓		
14:05			V	212 A 212 B							
14:06			Sy	126 A 126 A	(Unused)						
14:08			Bag	69	89	65	146	115		—	
14:09			V	213 A 213 B							
				END	@ 14:09						
	AW-4	14:23									
14:28	↓	↓	Bag	70	92	65	145	114	20.0	.2	
14:31			Bag	71	91	65	148	113	↓	.1	

4 of 4

ETHYLENE OXIDE SAMPLING DATA SHEET

DATE 6/23/88
RUN ID 3
SAMPLER PTL/WPG

SCHEDULE:
INITIAL EXHAUST 1
AIR WASHES 5

FACILITY Burron
BAROMETRIC PRESSURE

TIME	EXHAUST #	START	CONTAINER	ID	TEMPERATURE (F)				PERCENT OXYGEN	Ps (IN Hg)	NOTES
					VENT	t'	PUMP	HEAT TRACE			
14:32	AW4	14:23	V	214A 214B							
14:33	↓	↓	Sy	127A 128A	} unused						
14:34			Bag	72	92	66	145	114	20.5	—	
14:35			V	215A 215B							

APPENDIX F

CHAMBER TEMPERATURE AND PRESSURE DATA

```
                 ELAPSED       GAS          CHAMBER       CHAMBER
                 TIME          TEMP         TEMP          PRESS
                 (MIN)         (C)          (C)           (PSIA)
                    8.7 ✓        35.3 ✓       44.0 ✓         7.3 ✓
IZE                 9.2 ✓        35.3 ✓       44.5 ✓         6.9 ✓
```

```
12/88 CYCLE PARAMETERS:
DATE :  6 / 21 / 88
TIME :    :    :
CODE :  # Run #1

PREVAC
  SPL                           2.0  P
HUMIDITY
  STEAM   LEVEL                 3.1  P
  DWELL  TIME                  15.0  M
CHARGE
  ETO   LEVEL                  23.7  P
STERILIZE
  TIME                         15.0  M
  TEMP                         52.0  C
POSTVAC  AIR
  SPL                           2.0  P
  SPH                          10.0  P
ALARMS
  HIGH  PRESS                  25.0  P
  HIGH  TEMP                   60.0  C
```

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E/ M	ELAPSED TIME (MIN)	GAS TEMP (C)	CHAMBER TEMP (C)	CHAMBER PRESS (PSIA)
AC	19.9	33.9	50.2	14.4
	20.0	34.0	50.0	14.0
	22.0	34.0	47.5	11.1
	24.0	34.0	45.7	8.9
	26.0	34.0	45.6	7.1
	28.0	34.1	46.3	5.7
	30.0	34.2	46.2	4.6
	32.0	34.3	46.8	3.8
	34.0	34.4	47.7	3.4
	36.0	34.4	47.9	3.0
	38.0	34.5	48.3	2.5
	40.0	34.6	48.8	2.3
	42.0	34.6	49.5	2.1
DITY	42.7	34.6	49.1	2.0
	44.0	34.7	50.1	2.4
	46.0	34.8	50.6	2.6
	48.0	34.8	51.0	2.8
	50.0	34.9	58.6	3.0
	52.0	35.0	51.5	3.1
DITY	52.9	35.0	51.6	3.2
	54.0	35.0	51.4	3.1
	56.0	35.1	51.4	3.1
	58.0	35.1	51.3	3.1
	60.0	35.1	51.5	3.1
	62.0	35.2	51.4	3.1
	64.0	35.2	51.5	3.1
	66.0	35.3	51.7	3.1
SE	68.0	52.5	51.8	3.2
	70.0	51.6	52.8	4.6
	72.0	50.3	53.6	6.2
	74.0	49.2	54.2	7.6
	76.0	47.7	54.4	8.9
	78.0	46.0	54.5	10.4
	80.0	46.1	54.5	11.8
	82.0	46.5	54.4	13.3
	84.0	45.3	54.6	14.8
	86.0	45.4	54.6	16.2
	88.0	44.6	54.4	17.6
	90.0	43.4	54.2	19.0
	92.0	42.3	54.2	20.4
	94.0	40.7	54.2	21.8
	96.0	41.5	54.2	23.3
LIZE	96.7	41.8	54.0	23.8
	98.0	40.6	53.8	24.2
	100.0	40.9	53.2	24.2
	102.0	41.0	52.6	24.2
	104.0	41.1	52.4	24.2
	106.0	41.2	52.2	24.2

	108.0	41.1	[illegible]	[illegible]
	110.0	41.1	[illegible]	24.1
JST	111.6	41.0	54.4	24.1
	112.0	41.0	56.5	24.1
	114.0	40.9	56.7	22.7
	116.0	40.7	55.5	21.1
	118.0	40.6	55.4	19.6
	120.0	40.5	55.5	18.4
	122.0	40.4	55.8	[illegible]
	124.0	40.3	56.1	16.5
	126.0	40.1	56.4	15.9
	128.0	40.1	56.7	15.4
	130.0	40.0	[illegible]	15.1
	132.0	39.9	57.2	14.9
VAC AIR	133.6	39.8	51.4	14.7
	134.0	39.8	51.4	14.3
	136.0	39.7	50.0	12.3
	138.0	39.6	49.4	10.7
	140.0	39.5	49.3	9.2
	142.0	39.5	49.4	8.0
	144.0	39.4	49.6	7.0
	146.0	39.4	49.6	6.1
	148.0	39.4	49.7	5.2
	150.0	39.4	49.9	4.6
	152.0	39.4	50.1	4.1
	154.0	39.3	50.3	3.7
	156.0	39.3	50.8	3.3
	158.0	39.3	50.9	3.1
	160.0	39.3	51.1	2.8
	162.0	39.3	51.2	2.5
	164.0	39.3	51.5	2.3
	166.0	39.3	51.5	2.2
	168.0	39.3	51.7	2.0
VAC AIR	168.6	39.3	59.2	2.0
	170.0	39.4	60.7	3.2
	172.0	39.4	61.8	5.0
	174.0	39.4	62.4	6.7
	176.0	39.4	62.4	8.2
	178.0	39.4	57.1	10.1
VAC AIR	178.2	39.4	57.0	10.0
	180.0	39.3	53.4	8.5
	182.0	39.2	51.0	6.9
	184.0	39.2	50.4	5.7
	186.0	39.2	50.2	4.7
	188.0	39.1	50.4	4.0
	190.0	39.1	50.7	3.5
	192.0	39.2	51.1	3.0
	194.0	39.2	51.6	2.7
	196.0	39.2	52.4	2.4
	198.0	39.2	52.5	2.2
VAC AIR	199.9	39.2	58.4	2.1
	200.0	39.2	58.3	2.3
	202.0	39.2	60.5	4.1
	204.0	39.3	61.5	5.8
	206.0	39.3	61.8	7.3
	208.0	39.3	61.9	8.8
VAC AIR	209.4	39.2	57.4	10.0
	210.0	39.2	55.7	9.5
	212.0	39.2	52.5	7.8
	214.0	39.1	50.8	6.3
	216.0	39.1	51.0	5.1
	218.0	39.1	50.3	4.1
	220.0	39.0	50.7	3.6
	222.0	39.0	52.2	3.1
	224.0	39.0	51.4	2.7

```
          110.0      41.1      52.1      24.1
JST       111.8      41.0      54.4      24.1
          112.0      41.0      56.5      24.1
          114.0      40.9      56.7      22.9
          116.0      40.7      55.5      21.1
          118.0      40.6      55.4      19.6
          120.0      40.5      55.5      18.4
          122.0      40.4      55.8      17.4
          124.0      40.3      56.1      16.5
          126.0      40.1      56.4      15.9
          128.0      40.1      56.7      15.4
          130.0      40.0      57.0      15.1
          132.0      39.9      57.2      14.9
AC AIR    133.8      39.8      51.4      14.7
          134.0      39.8      51.4      14.3
          136.0      39.7      50.0      12.3
          138.0      39.6      49.4      10.7
          140.0      39.5      49.3       9.2
          142.0      39.5      49.4       8.0
          144.0      39.4      49.6       7.0
          146.0      39.4      49.6       6.1
          148.0      39.4      49.7       5.2
          150.0      39.4      49.9       4.6
          152.0      39.4      50.1       4.1
          154.0      39.3      50.9       3.7
          156.0      39.3      50.8       3.3
          158.0      39.3      50.9       3.1
          160.0      39.3      51.1       2.8
          162.0      39.3      51.2       2.5
          164.0      39.3      51.5       2.3
          166.0      39.3      51.5       2.2
          168.0      39.3      51.7       2.0
AC AIR    168.6      39.3      59.2       2.0
          170.0      39.4      60.7       3.2
          172.0      39.4      61.8       5.0
          174.0      39.4      62.4       6.7
          176.0      39.4      62.4       8.2
          178.0      39.4      57.1      10.1
AC AIR    178.2      39.4      57.0      10.0
          180.0      39.3      53.4       8.5
          182.0      39.2      51.0       6.[illegible]
          184.0      39.2      50.4       5.7
          186.0      39.2      50.2       4.7
          188.0      39.1      50.4       4.0
          190.0      39.2      50.7       3.5
          192.0      39.2      51.1       3.0
          194.0      39.2      51.8       2.7
          196.0      39.2      52.4       2.4
          198.0      39.2      52.5       2.2
AC AIR    199.9      39.2      58.4       2.1
          200.0      39.2      58.3       2.3
          202.0      39.2      60.5       4.1
          204.0      39.3      61.5       5.8
          206.0      39.3      61.8       7.3
          208.0      39.3      61.9       8.8
AC AIR    209.4      39.2      57.4      10.0
          210.0      39.2      55.7       9.5
          212.0      39.2      52.5       7.8
          214.0      39.1      50.8       6.3
          216.0      39.1      51.0       5.1
          218.0      39.1      50.3       4.1
          220.0      39.0      50.7       3.6
          222.0      39.0      52.2       3.1
          224.0      39.0      51.4       2.7
       →  226.0      39.0      51.6       2.4
```

Time wrong
Printer Jam

12.0 probably = 228.0

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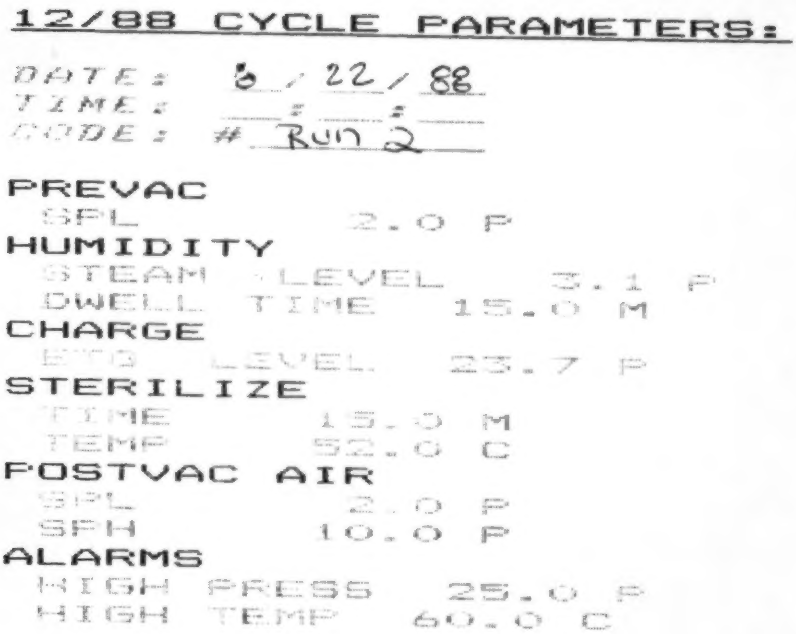
```
                      39.1                   51.5                    2.1
IR    [illegible]               39.1                   51.9                   2.0
[illegible]           39.1                   51.6                    2.7
14.0                  39.1                   55.0                    4.2
18.0                  39.2                   56.1                    6.0
[illegible]           39.3                   62.6                    7.7
22.0                  39.3                   62.7                    9.2
IR    33.1                      39.3                   56.8                   10.0
[illegible]           39.2                   55.5                    9.3
26.0                  39.2                   52.7                    7.5
29.0                  39.2                   50.8                    6.0
[illegible]           39.2                   50.6                    4.8
[illegible]           39.2                   50.1                    3.9
34.0                  39.2                   50.[illegible]          3.4
[illegible]           39.2                   51.0                    3.0
[illegible]           39.2                   51.2                    2.6
[illegible]           39.2                   51.5                    2.3
[illegible]           39.2                   51.7                    2.1
IR    [illegible]               39.3                   59.0                   2.1
44.0                  39.2                   60.8                    3.3
[illegible]           39.3                   62.4                    4.8
48.[illegible]        39.3                   62.7                    5.9
[illegible]           39.3                   63.1                    8.2
[illegible]           39.2                   62.8                    9.7
IR    51.4                      39.2                   57.2                   10.0
54.[illegible]        39.1                   54.4                    8.[illegible]
[illegible]           39.1                   [illegible]             5.9
[illegible]           39.[illegible]         [illegible]             5.8
[illegible]           [illegible]            [illegible]             4.1
[illegible]           39.[illegible]         50.9                    3.7
[illegible]           39.0                   50.7                    3.2
[illegible]           39.0                   51.0                    2.8
[illegible]           39.1                   51.6                    2.5
[illegible]           39.2                   51.5                    2.2
[illegible]           39.2                   51.7                    2.0
IR    [illegible]               39.[illegible]         57.[illegible]         2.1
[illegible]           39.[illegible]         60.[illegible]          3.8
[illegible]           39.3                   61.5                    5.4
[illegible]           39.4                   61.8                    6.9
[illegible]           39.5                   61.9                    8.7
80.0                  39.6                   61.7                   10.2
[illegible]           39.6                   61.4                   11.8
86.0                  39.6                   61.0                   13.2
87.5                  39.6                   60.7                   13.7
```



```
12/88 CYCLE PARAMETERS:
DATE:  6 / 22 / 88
TIME:  ___:___:___
CODE:  # Run 2
PREVAC
 SPL          2.0 P
HUMIDITY
 STEAM  LEVEL       3.1 P
 DWELL  TIME   15.0 M
CHARGE
 ETO  LEVEL    23.7 P
STERILIZE
 TIME        15.0 M
 TEMP        52.0 C
POSTVAC AIR
 SPL          2.0 P
 SPH         10.0 P
ALARMS
 HIGH PRESS  25.0 P
 HIGH TEMP  60.0 C
```

ASE/ LARM	ELAPSED TIME (MIN)	GAS TEMP (C)	CHAMBER TEMP (C)	CHAMBER PRESS (PSIA)
EVAC	0.2	35.0	53.9	14.6
	2.0	35.0	52.7	13.8
	4.0	34.9	49.5	10.9
	6.0	34.9	48.4	8.8
	8.0	34.9	48.2	6.9
	10.0	34.9	48.3	5.6
	12.0	34.9	48.5	4.5
	14.0	34.9	49.2	3.8
	16.0	35.0	49.8	3.4
	18.0	35.1	50.1	3.0
	20.0	35.1	50.5	2.6
	22.0	35.1	50.8	2.4
	24.0	35.2	51.0	2.1
UMIDITY	25.6	35.2	59.2	2.0
	26.0	35.2	59.9	2.4
	28.0	35.3	60.7	2.7
	30.0	35.4	61.2	3.0
UMIDITY	31.3	35.4	60.5	3.1
	32.0	35.4	53.6	3.1
	34.0	35.4	54.0	3.0
	36.0	35.4	52.7	3.1
	38.0	35.4	56.6	3.0
	40.0	35.4	52.6	3.0
	42.0	35.5	52.6	3.0
	44.0	35.5	52.7	3.0
	46.0	35.6	52.8	3.0
HARGE	46.2	35.6	60.5	3.0
	48.0	56.2	53.5	4.4
	50.0	57.6	54.3	5.7
	52.0	58.3	56.3	7.2
	54.0	58.7	62.3	8.6
	56.0	58.5	56.3	10.2
	58.0	58.6	55.4	11.6
	60.0	58.6	55.4	12.9
	62.0	58.3	55.4	14.4
	64.0	57.5	55.4	15.6
	66.0	57.3	57.4	17.2
	68.0	56.8	56.3	18.6
	70.0	56.4	56.2	20.2
	72.0	56.1	55.5	21.4
	74.0	55.2	55.2	23.0
TERILIZE	75.3	55.2	55.9	23.8
	76.0	54.8	55.2	24.3
	78.0	54.7	54.2	24.2
	80.0	54.4	53.7	24.1
	82.0	54.0	53.4	24.1
	84.0	53.4	57.9	24.1
	86.0	52.6	57.8	24.0
	88.0	52.3	57.3	24.1
	90.0	51.7	57.0	24.0
HAUST	90.2	51.6	57.2	24.0
	92.0	51.1	56.7	[illegible]

	96.0	50.1	56.2	19.2
	98.0	49.6	56.3	18.1
	100.0	49.1	56.5	17.2
	102.0	48.7	56.7	16.4
	104.0	48.2	57.1	15.8
	106.0	47.6	57.2	15.4
	108.0	47.4	57.5	15.0
	110.0	47.0	57.8	14.8
STVAC AIR	111.1	46.8	51.9	14.7
	112.0	46.6	51.5	13.7
	114.0	46.2	50.2	11.9
	116.0	45.9	49.9	10.3
	118.0	45.5	49.8	8.9
	120.0	45.2	49.9	7.8
	122.0	44.9	49.9	6.7
	124.0	44.6	50.0	5.8
	126.0	44.3	50.2	5.1
	128.0	44.0	50.4	4.5
	130.0	43.8	50.6	4.0
	132.0	43.5	50.7	3.6
	134.0	43.3	51.1	3.3
	136.0	43.1	51.3	3.0
	138.0	43.0	51.3	2.8
	140.0	42.7	51.6	2.5
	142.0	42.6	51.6	2.3
	144.0	42.5	51.6	2.1
	146.0	42.3	51.8	2.0
STVAC AIR	146.2	42.3	60.2	2.1
	148.0	42.2	61.7	3.3
	150.0	42.1	62.5	5.2
	152.0	42.0	62.9	6.8
	154.0	41.9	63.0	8.5
	156.0	41.8	62.8	10.0
STVAC AIR	156.1	41.8	55.7	10.1
	158.0	41.6	53.3	8.6
	160.0	41.3	51.3	7.1
	162.0	41.2	50.6	5.8
	164.0	41.1	50.4	4.8
	166.0	41.0	50.4	4.0
	168.0	40.8	50.9	3.5
	170.0	40.8	51.1	3.1
	172.0	40.6	51.4	2.8
	174.0	40.6	51.6	2.4
	176.0	40.5	51.7	2.2
	178.0	40.4	51.9	2.0
STVAC AIR	178.1	40.5	59.8	2.0
	180.0	40.4	61.4	3.5
	182.0	40.5	62.6	5.2
	184.0	40.4	63.1	6.7
	186.0	40.4	63.0	8.6
	188.0	40.3	57.1	10.0
STVAC AIR	188.1	40.3	56.1	10.0
	190.0	40.2	53.6	8.5
	192.0	40.1	51.0	6.8
	194.0	40.0	50.2	5.5
	196.0	40.0	50.1	4.4
	198.0	40.0	50.3	3.7
	200.0	39.9	50.8	3.2
	202.0	39.9	51.0	2.9
	204.0	39.9	51.2	2.5
	206.0	39.9	51.5	2.3
	208.0	39.9	51.7	2.0
STVAC AIR	208.5	39.9	59.3	2.0
	210.0	39.9	60.7	3.1
	212.0	40.0	62.3	4.6

	216.0	39.9	63.0	8.0
	218.0	39.9	62.7	9.7
STVAC AIR	218.5	39.9	56.8	10.0
	220.0	39.9	54.5	9.1
	222.0	39.8	51.3	7.5
	224.0	39.8	50.4	6.0
	226.0	39.7	50.0	4.8
	228.0	39.8	50.0	4.0
	230.0	39.8	50.4	3.3
	232.0	39.8	50.8	3.0
	234.0	39.8	51.0	2.6
	236.0	39.9	51.4	2.4
	238.0	39.9	51.9	2.1
STVAC AIR	239.1	39.9	58.6	2.1
	240.0	39.9	59.2	2.7
	242.0	40.0	61.5	4.3
	244.0	40.0	62.3	5.9
	246.0	40.0	62.5	7.5
	248.0	40.0	62.5	9.3
STVAC AIR	249.1	40.0	56.5	10.1
	250.0	40.0	55.5	9.3
	252.0	39.9	51.9	7.5
	254.0	39.9	50.3	6.1
	256.0	39.9	49.9	4.9
	258.0	39.9	50.1	3.9
	260.0	40.0	50.4	3.4
	262.0	40.0	50.8	3.0
	264.0	40.0	51.4	2.6
	266.0	40.0	51.5	2.3
	268.0	40.0	51.7	2.1
STVAC AIR	268.9	40.0	59.6	1.9
	270.0	40.1	60.8	3.1
	272.0	40.1	62.8	4.7
	274.0	40.2	63.4	6.1
	276.0	40.2	63.5	7.8
	278.0	40.1	63.4	9.6
STVAC AIR	278.7	40.1	56.7	9.9
	280.0	40.1	54.8	9.0
	282.0	40.1	51.7	7.1
	284.0	40.0	50.2	5.7
	286.0	40.1	49.9	4.6
	288.0	40.0	50.4	3.8
	290.0	40.0	50.8	3.3
	292.0	40.1	50.8	2.9
	294.0	40.0	51.2	2.5
	296.0	40.1	51.4	2.3
	298.0	40.1	51.6	2.0
STVAC AIR	298.3	40.1	59.6	2.0
	300.0	40.2	61.9	3.6
	302.0	40.2	63.1	5.2
	304.0	40.2	63.8	6.7
	306.0	40.3	63.6	8.4
	308.0	40.3	63.5	10.1
	310.0	40.3	63.1	11.5
	312.0	40.3	62.8	13.0
	313.7	40.4	62.3	13.9

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                  TIME: 6:30 :Am
PROGRAM #4 PARAMETERS
EVACUATE
SPL.........     1.5 PSI                -23-88
HUMIDITY
SPH.........     2.6 PSI
DWELL.......    30.0 MINUTES
CHARGE
NIT SPH.....     6.7 PSI
ETO SPH.....    12.1 PSI
NIT CAP.....    13.0 PSI
STERILIZE
TIME........   125.0 MINUTES
TEMP........    52.0 CENTIGRADE
PRE/POST NITROGEN PURGE
SPH.........    13.0 PSI
# OF CYCLES.     1.0
POST VAC AIR
SPH.........    10.0 PSI
# OF CYCLES.     4.0
ALARMS
HI PRESS....    14.0 PSI
HI TEMP.....    60.0 CENTIGRADE
```

	CYCLE TIME (MIN)	GAS TEMP (C)	CHMBR TEMP (C)	CHMBR PRESS (PSI)
PRE-VAC	0.0	37.0	43.8	14.2
	2.0	36.9	45.0	13.5
	4.0	36.9	44.9	11.3
	6.0	36.8	44.7	9.4
	8.0	36.7	44.7	7.5
	10.0	36.7	44.7	5.7
	12.0	36.7	45.1	4.4
	14.0	36.6	45.7	3.4
	16.0	36.6	46.3	2.7
	18.0	36.6	47.0	2.1
	20.0	36.6	47.5	1.8
LEAK TEST	21.5	36.6	47.7	1.5
	22.0	36.7	47.7	1.5
	24.0	36.7	48.5	1.5
	26.0	36.7	49.1	1.6
	28.0	36.7	49.6	1.6
	30.0	36.7	50.1	1.6
PRE-NITROGEN PURGE	31.4	36.8	50.3	1.6
	32.0	36.4	50.3	1.8
	34.0	38.0	50.1	2.2
	36.0	40.7	50.1	2.7
	38.0	43.9	50.1	3.2
	40.0	47.2	50.3	3.7
	42.0	50.4	50.6	4.2

RE-NITROGEN PURGE	31.4	36.8	50.3	1.6
	32.0	36.4	50.3	1.8
	34.0	38.0	50.1	2.2
	36.0	40.7	50.1	2.7
	38.0	43.9	50.1	3.2
	40.0	47.2	50.3	3.7
	42.0	50.4	50.6	4.2
	44.0	53.2	50.8	4.7
	46.0	55.6	51.0	5.2
	48.0	57.6	51.3	5.7
	50.0	59.2	51.4	6.2
	52.0	60.4	51.6	6.7
	54.0	61.4	51.8	7.2
	56.0	62.2	51.9	7.7
	58.0	62.8	52.0	8.2
	60.0	63.2	52.1	8.7
	62.0	63.4	52.2	9.2
	64.0	63.7	52.2	9.7
	66.0	63.8	52.3	10.2
	68.0	63.9	52.3	10.7
	70.0	64.0	52.3	11.2
	72.0	64.0	52.3	11.7
	74.0	64.1	52.3	12.2
	76.0	64.1	52.3	12.7
	78.0	63.2	52.2	13.0
	80.0	60.6	51.1	11.2
	82.0	58.8	48.9	9.3
	84.0	57.5	47.9	7.3
	86.0	56.4	47.5	5.6
	88.0	55.5	47.6	4.3
	90.0	54.8	48.0	3.4
	92.0	54.1	48.5	2.6
	94.0	53.5	49.0	2.1
	96.0	53.0	49.3	1.8
UMIDIFICATION	97.6	52.6	49.5	1.5
	98.0	52.5	49.7	1.8
	100.0	52.0	51.0	2.0
	102.0	51.6	52.1	2.2
	104.0	51.2	52.9	2.3
	106.0	50.8	53.4	2.4
	108.0	50.5	54.0	2.5
	110.0	50.1	54.3	2.5
HUMIDITY DWELL	110.1	50.1	54.2	2.6
	112.0	49.7	54.2	2.5
	114.0	49.3	54.1	2.5
	116.0	49.0	54.0	2.4
	118.0	48.7	54.1	2.4
	120.0	48.4	54.0	2.4
	122.0	48.0	53.9	2.4
	124.0	47.8	53.9	2.4
	126.0	47.5	53.8	2.4
	128.0	47.2	53.8	2.4
	130.0	46.9	53.7	2.4
	132.0	46.7	53.7	2.4
	134.0	46.4	53.6	2.4
	136.0	46.1	53.6	2.4
	138.0	45.9	53.5	2.4
NITROGEN CHARGE	139.9	45.7	53.5	2.4

	132.0	46.7	53.7	2.4
	134.0	46.4	53.6	2.4
	136.0	46.1	53.6	2.4
	138.0	45.9	53.5	2.4
NITROGEN CHARGE	139.9	45.7	53.5	2.4
	140.0	45.7	53.5	2.4
	142.0	47.3	53.3	2.8
	144.0	49.1	53.4	3.3
	146.0	51.1	53.5	3.8
	148.0	53.2	53.6	4.3
	150.0	55.2	53.6	4.8
	152.0	57.1	53.7	5.3
	154.0	58.7	53.8	5.8
	156.0	60.0	53.8	6.3
	158.0	60.1	53.9	6.7
	160.0	57.4	53.6	6.7
	162.0	55.7	53.4	6.7
	164.0	54.5	53.3	6.7
	166.0	53.6	53.2	6.7
ETO CHARGE	167.7	53.1	53.2	6.7
	168.0	51.5	53.2	6.7
	170.0	58.8	53.4	7.6
	172.0	60.6	53.8	8.2
	174.0	61.6	54.2	9.0
	176.0	61.4	54.4	9.6
	178.0	60.6	54.5	10.3
	180.0	59.3	54.4	11.1
	182.0	58.3	54.4	11.8
NITROGEN CAP	183.1	58.0	54.4	12.1
	184.0	37.9	53.6	12.6
	186.0	39.5	52.7	13.0
EXPOSURE	186.1	39.7	52.7	13.0
	188.0	37.5	52.7	12.9
	190.0	36.4	52.7	12.9
	192.0	36.2	52.7	12.9
	194.0	36.3	52.8	12.8
	196.0	36.6	52.8	12.8
	198.0	36.8	52.8	12.8
	200.0	37.0	52.8	12.8
	202.0	37.2	52.8	12.7
	204.0	37.4	52.8	12.7
	206.0	37.5	52.8	12.7
	208.0	37.6	52.8	12.7
	210.0	37.7	52.8	12.7
	212.0	37.8	52.8	12.7
	214.0	37.9	52.7	12.6
	216.0	38.0	52.5	12.6
	218.0	38.1	52.4	12.6
	220.0	38.2	52.5	12.6
	222.0	38.3	52.5	12.6
	224.0	38.4	52.5	12.6
	226.0	38.4	52.5	12.6
	228.0	38.5	52.4	12.6
	230.0	38.6	52.4	12.5
	232.0	38.7	52.3	12.5
	234.0	38.8	52.2	12.5
	236.0	38.8	52.3	12.5
	238.0	38.8	52.2	12.5

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240.0	38.9	52.2	12.5
242.0	39.0	52.2	12.5
244.0	39.0	52.2	12.5
246.0	39.1	52.2	12.5
248.0	39.2	52.3	12.5
250.0	39.3	52.5	12.4
252.0	39.3	52.2	12.4
254.0	39.3	52.1	12.4
256.0	39.4	52.0	12.4
258.0	39.4	51.9	12.4
260.0	39.4	51.9	12.4
262.0	39.4	51.8	12.4
264.0	39.5	51.9	12.4
266.0	39.4	51.9	12.4
268.0	39.5	51.8	12.4
270.0	39.5	51.8	12.4

	272.0	39.5	51.9	12.4
	274.0	39.5	51.9	12.4
	276.0	39.5	51.8	12.4
	278.0	39.6	51.8	12.4
	280.0	39.6	51.8	12.4
	282.0	39.6	51.9	12.4
	284.0	39.6	51.8	12.3
	286.0	39.6	51.8	12.3
	288.0	39.6	51.8	12.3
	290.0	39.6	51.8	12.3
	292.0	39.6	51.8	12.3
	294.0	39.6	51.8	12.3
	296.0	39.7	51.8	12.3
	298.0	39.7	51.8	12.3
	300.0	39.6	51.8	12.3
	302.0	39.6	51.8	12.3
	304.0	39.6	51.7	12.3
	306.0	39.6	51.8	12.3
	308.0	39.6	51.8	12.3
	310.0	39.7	51.8	12.3
POST-NITROGEN PURGE	310.0	39.7	51.8	12.3
	312.0	39.7	51.7	11.6
	314.0	39.7	50.7	9.6
	316.0	39.6	49.8	7.6
	318.0	39.6	49.2	6.0
	320.0	39.6	49.0	4.8
	322.0	39.7	49.1	3.9
	324.0	39.8	49.3	3.2
	326.0	39.8	49.4	2.7
	328.0	39.8	49.7	2.3
	330.0	39.9	49.9	2.0
	332.0	39.9	50.1	1.8
	334.0	40.0	50.3	1.6
	336.0	40.1	50.4	1.8
	338.0	41.7	50.3	2.2
	340.0	44.0	50.4	2.7
	342.0	46.3	50.5	3.2
	344.0	48.7	50.8	3.8
	346.0	51.0	51.1	4.3
	348.0	53.1	51.3	4.8
	350.0	54.8	51.6	5.3
	352.0	56.1	51.8	5.8
	354.0	57.2	52.0	6.2

	346.0	51.0	51.1	4.3
	348.0	53.1	51.3	4.8
	350.0	54.8	51.6	5.3
	352.0	56.1	51.8	5.8
	354.0	57.3	52.0	6.3
	356.0	58.1	52.2	6.8
	358.0	58.8	52.3	7.3
	360.0	59.3	52.4	7.8
	362.0	59.7	52.5	8.3
	364.0	60.0	52.5	8.8
	366.0	60.2	52.5	9.3
	368.0	60.4	52.6	9.9
	370.0	60.7	52.6	10.4
	372.0	60.8	52.7	10.9
	374.0	60.7	52.7	11.4
	376.0	60.8	52.7	11.9
	378.0	60.9	52.7	12.4
	380.0	61.0	52.8	12.9
POST AIR PURGE	380.3	61.0	52.8	13.0
	382.0	59.3	52.6	12.4
	384.0	57.6	51.0	10.6
	386.0	56.3	49.5	8.6
	388.0	55.2	48.6	6.7
	390.0	54.4	48.4	5.2
	392.0	53.9	48.6	4.1
	394.0	53.2	48.9	3.2
	396.0	52.8	49.2	2.6
	398.0	52.4	49.4	2.1
	400.0	52.0	49.8	2.0
	402.0	51.7	50.3	2.1
	404.0	51.3	50.9	2.8
	406.0	51.1	52.2	5.0
	408.0	50.8	53.2	6.9
	410.0	50.5	53.9	8.8
	412.0	50.2	54.4	10.0
	414.0	49.8	52.2	8.2
	416.0	49.5	49.8	6.4
	418.0	49.2	48.9	5.0
	420.0	48.8	48.9	3.9
	422.0	48.5	49.0	3.1
	424.0	48.3	49.2	2.5
	426.0	48.1	49.6	2.0
	428.0	47.9	50.2	2.1
	430.0	47.8	50.5	2.1
	432.0	47.6	51.3	3.3
	434.0	47.5	52.7	5.4
	436.0	47.4	53.6	7.5
	438.0	47.2	54.1	9.3
	440.0	46.9	53.6	8.6
	442.0	46.7	50.7	6.7
	444.0	46.5	49.4	5.2
	446.0	46.3	48.9	4.0
	448.0	46.2	48.9	3.1
	450.0	46.0	49.2	2.5
	452.0	45.9	49.6	2.1
	454.0	45.8	50.2	2.0
	456.0	45.7	50.6	2.1
	458.0	45.6	51.3	3.3
	460.0	45.6	52.9	5.1

```
                        406.0      51.1      52.2       5.0
                        408.0      50.8      53.2       6.9
                        410.0      50.5      53.9       8.8
                        412.0      50.2      54.4      10.0
                        414.0      49.8      52.2       8.2
                        416.0      49.5      49.8       6.4
                        418.0      49.2      48.9       5.0
                        420.0      48.8      48.9       3.9
                        422.0      48.5      49.0       3.1
                        424.0      48.3      49.2       2.5
                        426.0      48.1      49.6       2.0
                        428.0      47.9      50.2       2.1
                        430.0      47.8      50.5       2.1
                        432.0      47.6      51.3       3.3
                        434.0      47.5      52.7       5.4
                        436.0      47.4      53.6       7.5
                        438.0      47.2      54.1       9.3
                        440.0      46.9      53.6       8.6
                        442.0      46.7      50.7       6.7
                        444.0      46.5      49.4       5.2
                        446.0      46.3      48.9       4.0
                        448.0      46.2      48.9       3.1
                        450.0      46.0      49.2       2.5
                        452.0      45.9      49.6       2.1
                        454.0      45.8      50.2       2.0
                        456.0      45.7      50.6       2.1
                        458.0      45.6      51.3       3.3
                        460.0      45.6      52.9       5.1
                        462.0      45.5      53.8       7.3
                        464.0      45.5      54.3       9.3
                        466.0      45.4      54.6      10.0
                        468.0      45.2      51.9       8.0
                        470.0      45.1      49.9       6.1
                        472.0      44.8      49.0       4.8
                        474.0      44.8      48.6       3.7
                        476.0      44.8      48.9       2.9
                        478.0      44.7      49.3       2.3
                        480.0      44.7      49.7       2.0
                        482.0      44.6      50.3       2.1
                        484.0      44.6      50.7       2.1
                        486.0      44.5      51.9       3.9
                        488.0      44.6      53.3       5.9
                        490.0      44.6      54.1       8.1
                        492.0      44.5      54.7       9.9
                        494.0      44.5      55.5      11.9
                        496.0      44.4      55.9      13.3
CYCLE COMPLETE          497.5      44.4      55.4      14.0




                          0.0      44.4      55.4      14.0
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